11. (Once amended) Filter aid according to claim [1] 8, [characterized in that] wherein the at least one other filter-active fraction [it] comprises kieselguhr.

12. (Once amended) Filter aid according to claim 1, [characterized in that] wherein the at least one other filter active fraction [it] comprises perlite.

13. (Once amended) Filter aid according to claim 1, [characterized in that the] wherein a mean particle dimension of the [ready-to-use] filter aid is below 3.0 mm.

14. (Once amended) Filter aid according to claim [1, characterized in that] 2, wherein [the] a mean fiber diameter of the wood fibers is below 1.0 mm [in the case of fibrous particles].

REMARKS

This is in response to the Office Action dated August 1, 2000 (Paper No. 8) Reexamination and reconsideration in light of the proceeding amendments and the following remarks are respectfully requested.

Substitute Specification

A substitute specification is enclosed with this response. As the Examiner will be aware, the instant application is the result of a filing under 37 USC § 371. The original specification was lodged in German and accordingly, a translation of same was required for examination purposes in the United States. The English language documents which were provided to the Examiner therefore consisted of two translations: one of the original specification and the other of the substitute pages. The use of two different fonts and point sizes in these two documents resulted in the mismatch noted by the Examiner in this Office Action.

In this response terms such as "hl" and "per t" have been changed to "hecto liter" and "per ton" respectively.

In order to assist in the Examiner's understanding of the revision process, the mark-up which the Examiner has requested, is presented in three stages. The first is a retyped version of the pages which is marked in red ink, the second is revision of the "retyped version" which takes the form of the substitute specification per se. The third is a redline, which is produced using software, is provided to show the changes which have been made to the "retyped version" in order to arrive at the final substitute specification per se.

The undersigned hereby states that no new matter is introduced into the substitute specification.

Claim Amendments

In this response, the claims have been revised to improve them in both terms of clarity and syntax. Claim 1 has been amended to make it clear that the finely divided wood particles which have had sensorially active substances removed, remain in the form of wood particles.

Rejections Under 35 USC § 102

1) The rejection of claims 1-2 and 8-9 rejected under 35 U.S.C. § 102(b) as being anticipated by U.S. Patent No. 5,866,241 to Tan et al., is traversed.

Rejections under the 35 USC § 102 statute, are based on the premise that to anticipate a claim, each and every element of the claim must be shown in a single reference. When a claimed element cannot be found in the reference, the reference does not anticipate the claimed invention. Further, it is incumbent upon the Examiner to identify where in the reference each element may be found. Ex parte Levy, 17 U.S.P.Q.2d 1461 (Bd. Pat. App. Intrf. 1990). Consequently, when the Examiner fails to identify a claimed element, the Examiner has failed to establish a prima facie case of anticipation.

This rejection is therefore traversed in that the claims call for wood particles as

different from wood pulp to be subjected to treatment with a dilute alkali solution at a temperature below 100°C and at atmospheric pressure, to a degree sufficient to remove the sensorially active substances from the wood particles and leave the wood particles as wood particles.

The rejection indicates that Tan et al. teach wood pulp fibers subjected to a dilute alkali metal salt solution at a temperature of from 15 to 60°C and including organic particulates and filter active fractions. However, wood pulp is the result of processing wood chips. This processing ranges from a) digestion under pressure followed by pressure release to separate the fiber content, to b) mechanical processing to achieve the same end. It therefore follows that further processing of wood pulp can only occur following a first pulp forming process. It is submitted that the processing which is set forth in the pending claims, does not in fact result in a pulp let alone a pulp which is subjected to further processing.

It is additionally submitted that the claims, by calling for a filter aid, differentiates over the absorbent material which is disclosed in the Tan et al. reference.

As established in ***In re Bullock,*** 604 F.2d 1362, 1365, 203 USPQ 171, 174 (CCPA 1979), the purpose set forth in the preambles of the claims themselves, is more than a mere statement of purpose; and that language is essential to particularly point out the invention defined by the claims. Further, in ***Perkin-Elmer Corp. v. Computervision Corp.,*** 732 F.2d 888, 896, 221 USPQ 669, 675 (Fed. Cir.), *cert. denied,* 469 U.S. 857 [225 USPQ 792] (1984) it was established that the limitations appearing in the preamble are necessary to give meaning to the claim and property defined by the invention.

Tan et al. discloses an *absorbent material* to be used as absorbent cores in articles such as disposable diapers, feminine hygiene products and incontinent devices (see column 1, lines 5-10). The absorbent core is typically formulated of a cellulosic wood fiber matrix or pipe, which pipe is capable of absorbing large quantities of fluid (column 1, lines 63-65).

The cellulosic fiber used in this absorbent core contains in one preferred embodiment wood pulp, at least a portion of which is produced by a process that includes the step of treating liquid suspension of pipe at a temperature of from 15°C to about 60°C with an aqueous alkali metal salt solution having an alkali metal salt concentration of from about 2 weight percent to about 25 weight percent of said solution for a period of time ranging from about 5 minutes to about 100 minutes (column 3, lines 66 to column 4, line 7). According to column 2, lines 43 to 48 this material satisfies the absorbency requirements needed for use as absorbent core in disposable absorbent articles and which simultaneously provides time and cost savings to both the pulp manufacturer and the manufacturer of the absorbent article.

The present invention, however, relates to an *ancillary filtering agent* specially for filtration purposes in beer production which is not as injurious to health and easier to dispose of than kieselguhr.

It is submitted that the products which result from the process disclosed in Tan et al. and that which result in accordance with claim 1 are different. In Tan et al. absorbency is paramount. This is fundamentally different from filtering out small amounts of material from a large volume of liquid such as is necessary during the production of beer (for example). Clearly, Tan et al. is not directed to producing a filter aid of the nature to which the pending claims are directed. In fact, it can be asserted that the absorbent material disclosed in Tan. et al. would tend to absorb considerable amounts of the product which is intended to be recovered.

Thus, apart from not disclosing the claimed material, Tan et al. does not provide any disclosure or suggestion of producing a filter aid simply by removing all sensorially effective substances from wood particles in order to produce an ancillary filtering agent for beer filtering purposes.

2) <u>The rejection of claims 1 to 12 under 35 USC 102(b) as being anticipated by or, in the alternative under 35 USC § 103(a), as being obvious over Hou et al. '462, is traversed</u>.

The Examiner's position that the patentability of a product does not depend on its method of production, and that if the product in a product-by-process claim is the same as or is obvious from a product of the prior art, then the claim is unpatenable even through the prior art product was made by a different process – is not well taken in this instance. In this situation, the product is different and process is at least in part similar. In other words, what is being claimed is a new product being produced using steps which are per se known. This is the reverse of the logic on which the rejection is based.

Hou et al. '462, discloses a filter media, comprising cellulose fiber as a matrix, and particulate filter aid, the surfaces of at least one of which have been modified with inorganic anionic colloidal silica, are free of extractable and free of discoloration (see column 3, lines 54 to 64). However, while cellulose can be extracted from wood chips, the claims are such as to require finely divided wood particles as different from cellulose fibers. It is therefore submitted that the cellulose fibers of the nature used in Hou et al. '462 are no longer wood particles and have been converted to a different material. The situation is similar to crude oil being used to anticipate gasoline. Gasoline is derived from crude oil however, the materials are not the same.

Further, the claimed invention is such that the surfaces of neither a matrix, nor a filter aid have been modified with inorganic anionic colloidal silica. Therefore, products known from Hou et al. '462 and that according to the present invention must be considered different.

3) The rejection of claims 1 to 14 under 35 USC § 102(b) as being anticipated by or, in the alternative under 35 USC § 103(a), as being obvious over U.S. Patent No. 4,488,969 to Hou, is traversed.

Hou '969 teaches to make a self supporting fibrons media sheet from a long, self-bonding structural fiber, to give the sheet sufficient structural integrity in both the wet "as formed," and in the final dried condition.

In case of the present invention finely divided wood particles like sawdust, sanding dust, wood shavings, wood chips, cutting waste, chipped wood, cotton, straw, hemp, flax, bast, grasses and the like is used as *filter aid* and not to form a *self-supporting fibrons matrix*. It is submitted that the materials are different for at least the reasons set forth above.

Further, the teachings of Hou '969 are such as lead the reader of ordinary skill away from any notion of using wood chips per se as a filter aid. A reference must be considered for all it teaches, including disclosures that teach away from the invention as well as disclosures that point toward the invention. ***Ashland Oil, Inc. v. Delta Resins & Refractories, Inc***. 776 F.2d 281, 227 U.S.P.Q. 657 (Fed. Cir. 1985). In fact, the process which is disclosed in Hou '969 is such that the product would tend to be unsuitable for a filter aid such as used in beer production. As established in in ***re Gordon***, 733 F.2d 900, 221 USPQ 1125 (Fed. Cir. 1984) - If the proposed modification would render the prior art invention being modified unsatisfactory for its intended purpose, then there is no suggestion or motivation to make the proposed modification.

4) The rejection of claim 1 under 35 USC § 102(b) as being anticipated by or, in the alternative under 35 USC § 103(a), as being obvious over U.S. Patent No. 4,599,240 to Thompson, is traversed.

Thompson discloses a process to provide highly purified cellulose which will satisfy the requirements of human consumption and use. While this highly purified cellulose can be used as filter aid, it is in fact pure cellulose as different from the wood particles from which it was extracted. That is to say, as disclosed at column 3, lines 12-38, it is only after being converted from the starting material via a number of separations, oxidizations and filterings, is the cellulose material ready for use as a filter aid. In other words, it is no longer the claimed "wood" particles. This reference therefore not only fails to disclose the claimed wood particles it teaches away from the claimed material and toward one which, due to the purification/treatments, is no longer wood.

It is therefore submitted that the Thompson reference does not disclose the claimed invention.

Further, it is again stressed that the Examiner's position with respect to the patentability of a product not depending on its method of production, is incorrect. Whether the product in a product-by-process claim is the same as or is obvious over a product of the prior art, is moot in this instance. In this situation, the product is different while the respective processes contain known steps. In other words, as pointed out supra, what is being claimed is a new product being produced using steps which are per se known.

Conclusion

It is submitted that the invention as now claimed is neither anticipated nor rendered obvious by the references which have been cited in this action. Favorable reconsideration and allowance of this application is courteously solicited.

Respectfully submitted,

Date: November 1, 2000

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Should additional fees be necessary in connection with the filing of this paper, or if a petition for extension of time is required for timely acceptance of same, the Commissioner is hereby authorized to charge Deposit Account No. 19-0741 for any such fees; and applicant(s) hereby petition for any needed extension of time.

SECOND STAGE OF MARK-UP

RETYPED TEXT OF FIRST STAGE MARK-UP AMENDED AS PER PRELIMINARY AMENDMENT FILED SEPT. 13, 1999

FILTER AID

The invention relates to a filter aid, as disclosed in the preamble of claim 1, a process for its production and its application.

Cellulose-based filter aids have been known for a long time ("Ullmann's Encyklopädie der Technischen Chemie", 3rd edition (1951), first volume, page 492, key word "felted layers" and page 493, key word "filter aids"). Cellulose is produced in a multi-step chemical process, in which all sensorially active materials are removed from the raw material.

Hence filter aids made of pure cellulose are used wherever the sensorial neutrality of the used filter aid is of great significance. Examples of cellulosic filter aids are EFC (low extract cellulose), fine powder cellulose, fine fibrillated cellulose, cationized powder cellulose, fine MCC (microcrystalline cellulose).

In contrast, filter aids made of untreated woodpulp are produced by mechanical comminution, thus only by physical treatment, and can, thus, release extractives (color, odor, flavor) during filtration. Therefore, the use of wood fiber-based filter aids is usually limited to industrial filtrations, where relatively little demand is placed on the sensory analysis.

Not only for filtration in the food and luxury food sector, but also for many industrial applications, they may not be considered, e.g. for sugar solutions (glucose, dextrose, fructose), molasses, dye solutions, fats and oils and the like.

The difficult field of beverage filtration demands, on the one hand, complete sensorial neutrality of the used filter aid; on the other hand, the number of commonly used filter aids are limited for economic reasons, since the maximum expense for the filter aid is fixed by the price of the mineral filter aids dominating this market.

Usually beer filtration take place in two steps. The first step usually involves a coarse filtration, during which operation the liquid usually passes through a precoated layer of a filter aid. This step is frequently followed by a fine filtration (membrane, kieselguhr, etc.).

The standard filter aid for the precoat-type filter in the beverage, especially beer, sector, is kieselguhr. A high percentage of the world beer production is clarified by kieselguhr filtration. Currently it exceeds more than 1.1 billion hl beer.

The total demand for filter aids is worldwide ca. 750,000 tons per year, where inorganic materials, like, for example, kieselguhr, perlite or bentonite, constitute by far the largest share of this amount. Of this total amount about 250,000 tons to 300,000 tons per year are consumed worldwide by the beverage industry, largely by the breweries, but also by producers of wine and fruit juices.

The amount of filter aids, which are based on organic raw materials that can regrow again (cellulose, woodpulp, etc.), is to date only ca. 20,000 tons per year, even though their use offers a plurality of advantages over inorganic filter aids.

Thus organic filter aids are natural materials, whose quality fluctuates only over a narrow range and whose occurrence can be renewed at regular intervals. In addition the use of organic filter aids presents neither a health risk nor harmful effects for the environment and nature. Pumps and conveying elements of the filtration system are protected as much as possible owing to the non-abrasive property. Finally the consumed filter cakes can be easily disposed, for example, through land management, composting or animal fodder.

Of course, organic filter aids are in part many times more expensive than kieselguhr or they exhibit filtration properties that do not completely match those of kieselguhr.

For this reason organic filter aids have not been able to prevail to date against kieselguhr or are used in any case together with kieselguhr (report by J. Speckner "Cellulose as Filter Aids" in the journal ("Brauwelt", vol. 124 (1984), issue 46, pages 2058 to 2066, in particular page 2062, left column top).

However, kieselguhr has become increasingly a problem. As a natural mineral material its occurrence is limited. Thus in the case of kieselguhr one must resort more and more to low grade qualities in order to meet the high demand of industry. The result is, however, a rising cost for the cleaning and processing of kieselguhr, which could in the long run have a negative impact on its economic situation.

An even greater impact presents, however, the fact that the users' attitude towards kieselguhr is becoming more critical.

This reservation stems from the problems posed to the lungs due to many natural mineral materials and hence kieselguhr, a feature that must be taken very seriously from the point of view of occupational medicine. In 1988 the World Health Organization (WHO) categorized kieselguhr as a carcinogenic substance following a series of animal

experiments. For handling there are strict regulations that are being asserted and observed more and more in Germany.

Another factor is that the disposal of kieselguhr is becoming increasingly more critical in industrial countries. A classification as hazardous waste makes landfilling considerably more difficult. With the introduction of the new technical rules on municipal waste, the disposal situation for kieselguhr is becoming more restrictive. In many cases, disposal of kieselguhr used as filter aid already gives rise to costs of approximately DM 600,000 per t of kieselguhr, if this was used in beer filtration, or 1500.00 per t of kieselguhr if this was used in the industrial filtration of problematic substances.

Starting from these urgent problems in particular in the beer filtration sector, the object underlying the invention is to develop a filter aid which is effective and can be provided economically.

This object is achieved by the invention described in claim 1.

Substances which are sensorially active, that is active with respect to color, odor and/or flavor, are to be removed from the filter aid to a sufficient extent prior to use as filter aid, so that none of these substances can transfer into the filtrate to a significant extent and impair its sensory properties. The particles are neutralized sufficiently to a certain extent sensorially in order to be able to serve as filter aid. It is essential here that the treatment is only carried out to the extent necessary for this purpose. The use of energy and chemicals remains in a justifiable range, so that the product can compete economically with kieselguhr. The action is therefore not so intensive as is the case in the production of cellulose from wood fibers. Surprisingly, it has been found that by means of a liquid treatment a sufficient neutralization of the particles with respect to sensory aspects can be achieved without the need for simultaneously high pressures and temperatures to be employed, large amounts of aggressive chemicals and treatment times of many hours to days. By means of the invention, to a certain extent an expanded area of application is open to the wood particles, without this needing expenditure as in the case of cellulose production.

Although the starting point and preferred area of application for the invention is beer and beverage filtration, which concerns the creation of a kieselguhr substitute material, the invention is not restricted to this area of application.

Although DE 23 51 125 A1 discloses using, for the extraction of metals or metal ions from solutions, an adsorbent which is obtained by bringing, inter alia, wood sawdust into contact with a solution of a hydroxide of an alkali metal or alkaline earth metal, this is an adsorption, i.e. an accumulation of dissolved particles on the surface of the adsorbent, not a filtration, i.e. a separation from a suspension.

In the case of DE 41 10 252 C1, for prefloating a filter cake, a mixture of filter aids of differing morphological and physical properties is used which comprise at least one component which increases the density of the filter cake and is made of high-density, chemically resistant metal and/or metal oxide and/or carbon particles of fibrous and/or granular structure and a further component of plastic fibers and/or cellulose fibers having a fiber length of from 1000 to 5000 μm and a fiber thickness of from 0.5 to 100 μm.

The wood particles of the filter aid according to the invention comprise, for example, wood fibers (claim 2) or, in particular, wood comminution residues (claim 3), that is for example sawdust, sanding dust, wood shavings, wood chips, cutting waste, chipped wood and the like.

The grinding performed in the production of the wood particles in the invention substantially determines the filtration properties. With fine grinding, the permeability of the filter layer is generally lower. By means of the grinding (micronization, fibrillation), in addition the particle shape is affected which in turn changes the water value, which is still to be explained, as a measure of the permeability of the filter layer. In the case of fibrous cellulose products these can be fibrilated to a greater or lesser extent. The grinding can also be performed in a plurality of steps in which a first grinding for the production of the particles is followed by a further grinding after the treatment and before or after the drying.

The filter aid particles produced from wood particles are still actually to have wood character, i.e. the lignin shall not have been virtually quantitatively extracted from the raw material wood, as occurs in cellulose production in the sulfite or sulfate process by treatment for many hours at elevated pressure at temperatures far above 100°C.

The treatment time in the invention can be relatively short, for example less than two hours, so that it is differentiated by one order of magnitude from the treatment time in the production of cellulose. The purpose is the removal, of only fractions of the wood which are unwanted with respect to the use as filter aid, i.e. have an effect in terms of flavor, odor and/or color in the filtrate. This is not in this case primarily lignin, but

compounds such as essential oils, terpeneoils and terpenoids, tannic acids, fats and waxes, phenolic substances (lignans, phenylpropanes, coumarin) stilbenes, flavonoids and the like, which make up an amount of from approximately 4 to 5 percent by weight of the dry wood. It has been found that these compounds can be, by means of a treatment with dilute alkali solutions acids [sic] even at room temperatures under atmospheric pressure, extracted from the wood or else made inactive to the extent that the treated wood particles are sufficiently neutral sensorially for the practical use as filter aid. It is not the case that during a rigorous analysis no residues of the unwanted type can be observed any longer, but that, for example, a medium filtered using the filter aid does not permit, during sensory testing, any wood flavor or wood aroma or any brown discoloration to be recognized. The treatment can be brief relative to the treatment durations of cellulose production.

An important feature in the treatment of the particles is in addition, that the treatment can also take place at temperatures below 100°C and simultaneously at atmospheric pressure, which substantially simplifies the plant required for producing the filter aid.

The filter aid of the invention can be prepared particularly economically. The costs may be in the same order of magnitude as the costs of kieselguhr, but only about one third of the costs for cellulose powder.

It also appears to be the case that the particles treated according to the invention have on the surface an additionally roughened or fissured structure which advantageously affects the filter properties.

Underlying the preamble of claim 1 is French Patent 385 035 which has, as its subject matter, wood fibers used for filter purposes which are treated, to avoid rotting, with a permanganate solution with or without addition of acid.

From JP-A 73 28 353, DE 41 10 252 C1 and EP 747 104 A2 can be taken the use of cellulose fibers that is not alkali-solution-treated wood particles mixed with other components as filter aid.

From FR 534 288 there arises a filter aid for wines, which aid consists of wood wool which is thoroughly washed and treated with 1% strength tartaric acid.

According to claim 4 the filter aid can comprise essentially only wood particles of one and the same type, size and pretreatment, that is can essentially be uniformly composed.

However, it is also possible according to claim 5 for the filter aid to comprise at least two particle fractions comminuted according to different processes, in order to be able to set the filtration properties in accordance with the requirements.

From the same aspect, the filter aid can comprise at least two particle fractions comminuted to different dimensions (claim 6) and/or at least two particles produced from different starting materials (claim 7).

The filter aid can also comprise other fractions which do not affect the filtration properties (claim 8).

It can also be a mixture with other filter-active constituents, i.e. not consisting of plant fibers (claim 9), also with mineral constituents (claim 10), namely with kieselguhr (claim 11), which would have the effect of reducing the kieselguhr content and the associated problems mentioned at the outset.

However, suitable additional constituents are also other mineral filter aids, in particular perlite (claim 12).

According to claim 13, the largest mean particle diameter of the ready-to-use filter aid shall be less than 3.0 mm.

In contrast, in the case of fibrous particles, the mean fiber diameter shall be less than 1.0 mm (claim 14).

Since the particles are produced by grinding, they do not have an exact size, but a size distribution for instance according to a Gaussian curve. The position of the maximum of this curve may be taken to mean here the highest particle dimension.

The filter aid of the invention can be used to form prefloat filter layers in the same manner as was previously the case with mineral filter aids.

The invention also extends to a process according to claim 15 for preparing the filter aid in which the particles are digested by the treatment liquid in the course of a period of action.

A suitable temperature range in the treatment of the particles is the range of room temperature, which, although it requires no heating energy consumption, does require longer treatment times (claim 16).

A further practicable region with shorter treatment times is 50-100°C (claim 17).

According to claim 18, atmospheric pressure in the temperature range of 70 to 90°C can be employed, which is a temperature markedly increased with respect to room

temperature, but is below the boiling point and eliminates the use of pressure vessels. This leads to a usable filter aid with a minimum of equipment and energy consumption.

"Dilute alkali solution" shall mean an aqueous solution having a content of from 2 to 10% by weight of the dry alkali, based on the solids content (claim 19).

In the preferred embodiment of the invention, sodium hydroxide solution is used (claim 20).

The period of action depends apart from the pressure and temperature, on the solution capacity of the dilute alkali solution for the unwanted constituents. In the case of dilute alkali solution, it is also the case that it is not periods of action in the seconds region which come into question, but those which are short in comparison with the periods of action of many hours to days necessary in cellulose production. The period of action is partly dependent on the particle size.

It is, moreover, of a size determined by the fact that precisely only the sensory-critical substances are to be removed from the particles, in particular the wood particles. The latter purpose is achieved when at most 10% by weight on an absolutely dry basis of the wood constituents are removed (claim 21), whereas the production of cellulose relates to the liberation of generally more than 30% of the wood constituents.

The period of action can be, with an alkali solution treatment, in particular between 5 and 120 min. (claim 22).

The consistency, i.e. the proportion by weight of the particles in the dilute alkali solution, can be from 5 to 25% in the treatment (claim 23).

The particles, after the period of action, can be washed and dried (claim 24).

The particle size (maximum of the particle size distribution) can be up to 10 mm, preferably 01 to 1.0 mm, during the treatment (claim 25).

Since a grinding in the wet phase changes the particle shape, a possibility is opened up in this manner of setting the water value (claim 26).

In individual cases it is possible, without relinquishing the lack of sensory hazard to further comminute the particles after the alkali solution treatment and the drying, simultaneously with the drying or after the drying (claim 27).

To obtain clear conditions with respect to the filter properties, it is advisable according to claim 28 to classify the particles after the alkali solution treatment and the drying.

The invention is also embodied in the use of finely divided wood particles which have been subjected to a treatment with a dilute lute [sic] at a temperature below 100°C and at atmospheric pressure, which treatment removes the sensorially active substances from the wood particles, as filter aid (claim 29), in particular when the particles have been treated according to the process of claims 15 to 28 (claim 30).

A suitable use is in particular in beverage filtration, in particular beer filtration (claim 31).

Other fields of application of the invention are food filtration (claim 32), for example sugar solutions, edible oil, fat, gelatin, citric acid, alginate etc., filtration in the chemical sector (claim 33), for example chloralkali, in the sector of the cleaning of auxiliary liquids in metalworking (claim 34), for example cutting fluids, rolling oils, polishing oils, etc., and in the pharmaceutical and cosmetics sector (claim 35).

To study the efficacy of the novel treatment of the plant fiber particles, untreated plant fiber particles (Lignocel C 120) were compared with plant fiber particles treated according to the invention (Sample No. 1; Sample No. 2; Sample No. 3). The Samples No. 1 to No. 3 were treated as follows:

Sample 1: To produce the treated plant fiber particles, 330 g of wood fiber flour (particle range: 70-150 Пm), 3700 ml of water and 15.8 g of solid sodium hydroxide were digested (reacted) in a mixing and treatment reactor at from 20°C to 25°C without additional heating and without stirring. The solids content was below 10% by weight, the retention time was at least 16 hours, the pH of the aqueous alkali solution was below 11.3 after 16 hours.

The sodium hydroxide solution was filtered off by vacuum via a plastic filter, the predried wet cake was slurried in hot water (70°C), so that a solids content below 15% by weight was achieved. A final pH of from 3.0 to 7.0 was set using dilute hydrochloric acid and the solution was filtered off under vacuum via a plastic filter. The subsequent rinsing was performed at least twice each time with 200 to 500 ml of water at 70°C.

Sample 2 was treated with hot alkali solution and rinsed cold. In a mixing and treatment reactor at temperatures above 50°C and with stirring 330 g of wood fiber flour (particle range: 70 - 150 μm), 3700 ml of water and less than 12 g solid sodium hydroxide were digested (reacted). The solids content was below 10% by weight; the retention time was at least 20 minutes; the pH value of the aqueous alkali solution was below 10.8 at the

end of the experiment. The sodium hydroxide solution was filtered off under vacuum via a plastic filter; the predried wet cake was slurried in hot water (70°C) so that a solids content below 15% by weight was achieved. A final pH ranging from 3.0 to 7.0 was set using dilute hydrochloric acid; and the solution was filtered off by vacuum via a plastic filter. The subsequent rinsing was performed at least twice each time with 200 to 500 ml of cold water at 20°C.

Sample 3 was produced at a pilot plant. The solids content was comparable with the laboratory batches. It was washed three times in cold water.

To determine the yield a thin layer of the resulting wet cake ranging from 5 to 10 mm was spread on a sheet and dried.

The degree of whiteness and the density was determined with this material.

The yield (on an absolutely dry basis) was at least 97% by weight, i.e. at most 3% by weight of the constituents of the wood fiber flour that was employed was released during the alkali solution treatment.

The sensorial test was conducted in an aqueous suspension, in which 1 g of product was suspended in 150 ml water at 100°C. With this suspension the smell and taste were tested.

To get an impression of what is still present in the untreated wood particle material (Lignocel C 120), on the one hand, and the wood particle material (sample nos. 1-3), subjected to the alkali solution treatment, the materials were subjected to an extraction in a Soxleth apparatus. The amount of the still extractive constituents present in the materials is a measure for the suitability of the material as a filter aid for sensorially challenging filtrations.

During the extraction operation in the Soxleth apparatus 5 g of the product dried to a moisture content below 10% by weight were extracted with 250 ml ethanol / water (1:1) for 5 hours; and the extract content was determined gravimetricly.

With the material dried to a moisture content below 10% by weight, a test filtration was subsequently conducted according to a procedure specified by the Schenk company at 20°C, in which procedure the wet cake height, the Darcy value, the fluming behavior, and the water value are determined.

The results of the test are compiled in the attached table.

The sensorial area was evaluated with numbers, where 0 denotes good, 10 denotes poor.

The table shows that the untreated material with respect to odor exhibits a value 8, which is obviously worse than the value of the treated samples nos. 1-3.

The same applies to the flavor, which was virtually impossible to evaluate with the untreated product Lignocel C 120.

An important point is the amount of extract. In the untreated product Lignocel C 120 3.37% could still be extracted, whereas the equivalent values of treated products were 1.0%. This means that the relatively mild alkali solution treatment had already released a noticeable amount of the extractable constituents, a feature that may be a disturbing factor in the use of the product as a filter aid.

The alkali solution treatment of the product and the subsequent washing operations make it possible to affect somewhat the water value, which is a measure for the permeability of the filter aid. The water value is determined with a laboratory pressure filter (diameter 50 mm) and an elevated water tank with level control. Between the level of the water in the elevated water tank and the filter bottom a difference of 2 m must be maintained.

The laboratory filter is provided and sealed with a moistened permeable layer of cellulose (Schenk D layer with the screen side downward). Then 25 g of filter aid are slurried in 200 to 300 ml pure water and completely transferred into the lab filter. The lab filter is attached to the elevated water tank and purged. After one minute 500 ml water are removed via a filter and then the time for the next 100 ml filtrate stopped. The water value follows from the stopped time as follows:

$$\text{water value} = \frac{480}{\text{time in minutes}}$$

If the result is a water value of less than 150, the determination is done as above, but with the use of only 4 g of filter aid. Then the result is:

$$\text{water value} = \frac{76.8}{\text{time in minutes}}$$

Hence the shorter the time, required for a specific volume of water to flow through the filter layer, the higher is the water value.

Table

Product	Moisture content % by weight	Odor	Flavor	Turbidity	Color	Extract Whiteness %	Bulk density g/dm^3	Wet cake height mm/25g	Darcy Value	Incoming flow behavior	Water value min^1/25g
Reference Lignocel C120	9.0	8	10 (bitter)	8 (yellow)	3.37	58.4	128	78	5.3	good	770
Sample No. 1	3.5	1	2-3 (mild)	1-2 (Colorless	0.93	34.2	Not determined	83	8.0	good	1098
Sample No. 2	5.3	1	2-3 (mild)	2 (Colorless	1.04	34.1	125	82	7.3	good	1010
Sample No. 3	7.5	5	6 (neutral)	2-3 (almost colorless)	0.98	36.8	131	79	7.8	good	1125

WE CLAIM:

1. Filter aid which comprises finely divided wood particles which have been subjected to a chemical liquid treatment, characterized in that the particles have been subjected to a treatment with a dilute alkali solution at a temperature below 100°C and at atmospheric pressure, which removes the sensorially active substances from the wood particles.

2. Filter aids according to claim 1, characterized in that the particles comprise wood fibers.

3. Filter aids according to claim 1, characterized in that the particles comprise wood comminution residues.

4. Filter aid according to one of claim 1, characterized in that it essentially comprises only wood particles of one and the same type, size distribution and pretreatment.

5. Filter aid according to one of claim 1, characterized in that it comprises at least two fractions of particles comminuted by different processes.

6. Filter aid according to one of claim 1, characterized in that it comprises at least two fractions of particles comminuted to different dimensions.

7. Filter aid according to one of claim 1, characterized in that it comprises fractions of particles produced from at least two different starting materials.

8. Filter aid according to one of claim 1, characterized in that it comprises other organic or inorganic fractions which do not affect the filtration properties.

9. Filter aid according to one of claim 1, characterized in that it comprises other filter-active fractions.

10. Filter aid according to one of claim 1, characterized in that it comprises other mineral fractions.

11. Filter aid according to one of claim 1, characterized in that it comprises kieselguhr.

12. Filter aid according to one of claim 1, characterized in that it comprises perlite.

13. Filter aid according to one of claim 1, characterized in that the mean particle dimension of the ready-to-use filter aid is below 3.0 mm.

14. Filter aid according to one of claim 1, characterized in that the mean fiber diameter is below 1.0 mm in the case of fibrous particles.

15. Process for producing the filter aid according to claim 1, characterized in that the particles are digested with the dilute alkali solution during a period of action.

16. Process according to claim 15, characterized in that the temperature of the dilute alkali solution during the treatment is in the range of room temperature.

17. Process according to claim 15, characterized in that the temperature of the dilute alkali solution during treatment is 50-100°C.

18. Process according to claim 15, characterized in that the temperature of the dilute alkali solution during the treatment is from 70 to 90°C.

19. Process according to claim 15, characterized in that concentration of the dilute alkali solution is from 2 to 10% by weight, based on the solids content.

20. Process according to claim 15, characterized in that the alkali solution used is sodium hydroxide solution.

21. Process according to claim 15, characterized in that the period of action is of a duration such that at most 10% by weight on an absolutely dry basis of the wood constituents are removed.

22. Process according to claim 15, characterized in that the period of action is from 5 to 120 min.

23. Process according to claim 15, characterized in that the consistency during the treatment is from 5 to 25%.

24. Process according to claim 15, characterized in that the particles are washed and dried after the period of action.

25. Process according to claim 15, characterized in that the particle size during the treatment is up to 10 mm, preferably from 0.1 to 1.0 mm.

26. Process according to claim 15, characterized in that the water value is set by influencing the grinding in the wet phase (refiner).

27. Process according to claim 15, characterized in that the particles are further comminuted after the treatment and before the drying, simultaneously with the drying or after the drying.

28. Process according to claim 15, characterized in that the particles are classified after the treatment and the drying.

29. The use of finely divided wood particles which have been subjected to a treatment with a dilute alkali solution at a temperature below 100°C and at atmospheric pressure, which treatment removes the sensorially active substances from the wood particles, as filter aid.

30. The use of finely divided wood particles which have been treated according to claim 15 as filter aid.

31. The use according to claim 29 in beverage filtration, in particular beer filtration.

32. The use according to claim 29 in food filtration.

33. The use according to claim 29 in the sector of the cleaning of liquids in the chemicals industry.

34. The use according to claim 29 in the sector of the cleaning of auxiliary liquids in metalworking.

35. The use according to claim 29 in the sector of pharmaceuticals and cosmetics.

ABSTRACT OF THE DISCLOSURE

The filter aid comprises finely divided plant fibers, which for a period of action have been subjected to a liquid treatment, which removes the sensorially active substances from the plant fibers.

THIRD STAGE OF MARK-UP

REDLINE COMPARING COMPLETED SUBSTITUTE SPECIFICATION WITH RETYPED TEXT

Titl of the Invention

FILTER AID

BACKGROUND OF THE INVENTION

Field of the Invention ~~[FILTER AID]~~

The invention relates to a filter aid, ~~[as disclosed in the preamble of claim 1,]~~ a process for its production and its application.

Description of the Related Art

Cellulose-based filter aids have been known for a long time ("Ullmann's Encyklopädie der Technischen Chemie", 3rd edition (1951), first volume, page 492, key word "felted layers" and page 493, key word "filter aids"). Cellulose is produced in a multi-step chemical process, in which all sensorially active materials are removed from the raw material.

Hence filter aids made of pure cellulose are used wherever the sensorial neutrality of the used filter aid is of great significance. Examples of cellulosic filter aids are EFC (low extract cellulose), fine powder cellulose, fine fibrillated cellulose, cationized powder cellulose, fine MCC (microcrystalline cellulose).

In contrast, filter aids made of untreated woodpulp are produced by mechanical comminution, thus only by physical treatment, and can, thus, release extractives (color, odor, flavor) during filtration. Therefore, the use of wood fiber-based filter aids is usually limited to industrial filtrations, where relatively little demand is placed on the sensory analysis.

Not only for filtration in the food and luxury food sector, but also for many industrial applications, they may not be considered, e.g. for sugar solutions (glucose, dextrose, fructose), molasses, dye solutions, fats and oils and the like.

The difficult field of beverage filtration demands, on the one hand, complete sensorial neutrality of the used filter aid; on the other hand, the number of commonly used filter aids are limited for economic reasons, since the maximum expense for the filter aid is fixed by the price of the mineral filter aids dominating this market.

Usually beer filtration take place in two steps. The first step usually involves a coarse filtration, during which operation the liquid usually passes through a precoated layer of a filter aid. This step is frequently followed by a fine filtration (membrane, kieselguhr, etc.).

The standard filter aid for the precoat-type filter in the beverage, especially beer, sector, is kieselguhr. A high percentage of the world beer production is clarified by kieselguhr filtration. Currently it exceeds more than 1.1 billion ~~[hl]~~ **hecto liter of** beer.

The total demand for filter aids is worldwide ca. 750,000 tons per year, where inorganic materials, like, for example, kieselguhr, perlite or bentonite, constitute by far the largest share of this amount. Of this total amount about 250,000 tons to 300,000 tons per year are consumed worldwide by the beverage industry, largely by the breweries, but also by producers of wine and fruit juices.

The amount of filter aids, which are based on organic raw materials that can regrow again (cellulose, woodpulp, etc.), is to date only ca. 20,000 tons per year, even though their use offers a plurality of advantages over inorganic filter aids.

Thus organic filter aids are natural materials, whose quality fluctuates only over a narrow range and whose occurrence can be renewed at regular intervals. In addition the use of organic filter aids presents neither a health risk nor harmful effects for the environment and nature. Pumps and conveying elements of the filtration system are protected as much as possible owing to the non-abrasive property. Finally the consumed filter cakes can be easily disposed, for example, through land management, composting or animal fodder.

Of course, organic filter aids are in part many times more expensive than kieselguhr or they exhibit filtration properties that do not completely match those of kieselguhr.

For this reason organic filter aids have not been able to prevail to date against kieselguhr or are used in any case together with kieselguhr (report by J. Speckner "Cellulose as Filter Aids" in the journal ("Brauwelt", vol. 124 (1984), issue 46, pages 2058 to 2066, in particular page 2062, left column top).

However, kieselguhr has become increasingly a problem. As a natural mineral material its occurrence is limited. Thus in the case of kieselguhr one must resort more and more to low grade qualities in order to meet the high demand of industry. The result is, however, a rising cost for the cleaning and processing of kieselguhr, which could in the long run have a negative impact on its economic situation.

An even greater impact presents, however, the fact that the users' attitude towards kieselguhr is becoming more critical.

This reservation stems from the problems posed to the lungs due to many natural mineral materials and hence kieselguhr, a feature that must be taken very seriously from the point of view of occupational medicine. In 1988 the World Health Organization (WHO) categorized kieselguhr as a carcinogenic substance following a series of animal experiments. For handling there are strict regulations that are being asserted and observed more and more in Germany.

Another factor is that the disposal of kieselguhr is becoming increasingly more critical in industrial countries. A classification as hazardous waste makes landfilling considerably more difficult. With the introduction of the new technical rules on municipal waste, the disposal situation for kieselguhr is becoming more restrictive. In many cases, disposal of kieselguhr used as filter aid already gives rise to costs of approximately DM 600,000 per ~~[t]~~ **ton** of kieselguhr, if this was used in beer filtration, or 1500.00 per ~~[t]~~ **ton** of kieselguhr if this was used in the industrial filtration of problematic substances.

SUMMARY OF THE INVENTION

Starting from these urgent problems in particular in the beer filtration sector, the object underlying the invention is to develop a filter aid which is effective and can be provided economically.

~~[This object is achieved by the invention described in claim 1.~~

~~]~~Substances which are sensorially active, that is active with respect to color, odor and/or flavor, are to be removed from the filter aid to a sufficient extent prior to use as filter aid, so that none of these substances can transfer into the filtrate to a significant extent and impair its sensory properties. The particles are neutralized

sufficiently to a certain extent sensorially in order to be able to serve as filter aid. It is essential here that the treatment is only carried out to the extent necessary for this purpose. The use of energy and chemicals remains in a justifiable range, so that the product can compete economically with kieselguhr. The action is therefore not so intensive as is the case in the production of cellulose from wood fibers. Surprisingly, it has been found that by means of a liquid treatment a sufficient neutralization of the particles with respect to sensory aspects can be achieved without the need for simultaneously high pressures and temperatures to be employed, large amounts of aggressive chemicals and treatment times of many hours to days. By means of the invention, to a certain extent an expanded area of application is open to the wood particles, without this needing expenditure as in the case of cellulose production.

Although the starting point and preferred area of application for the invention is beer and beverage filtration, which concerns the creation of a kieselguhr substitute material, the invention is not restricted to this area of application.

Although DE 23 51 125 Al discloses using, for the extraction of metals or metal ions from solutions, an adsorbent which is obtained by bringing, inter alia, wood sawdust into contact with a solution of a hydroxide of an alkali metal or alkaline earth metal, this is an adsorption, i.e. an accumulation of dissolved particles on the surface of the adsorbent, not a filtration, i.e. a separation from a suspension.

In the case of DE 41 10 252 C1, for prefloating a filter cake, a mixture of filter aids of differing morphological and physical properties is used which comprise at least one component which increases the density of the filter cake and is made of high-density, chemically resistant metal and/or metal oxide and/or carbon particles of fibrous and/or granular structure and a further component of plastic fibers and/or cellulose fibers having a fiber length of from 1000 to 5000 μm and a fiber thickness of from 0.5 to 100 μm.

The wood particles of the filter aid according to the invention comprise, for example, wood fibers ~~{(claim 2)}~~ or, in particular, wood comminution residues~~{(claim 3)}~~, that is for example sawdust, sanding dust, wood shavings, wood chips, cutting waste, chipped wood and the like.

The grinding performed in the production of the wood particles in the invention substantially determines the filtration properties. With fine grinding, the

permeability of the filter layer is generally lower. By means of the grinding (micronization, fibrillation), in addition the particle shape is affected which in turn changes the water value, which is still to be explained, as a measure of the permeability of the filter layer. In the case of fibrous cellulose products these can be fibrilated to a greater or lesser extent. The grinding can also be performed in a plurality of steps in which a first grinding for the production of the particles is followed by a further grinding after the treatment and before or after the drying.

The filter aid particles produced from wood particles are still actually to have wood character, i.e. the lignin shall not have been virtually quantitatively extracted from the raw material wood, as occurs in cellulose production in the sulfite or sulfate process by treatment for many hours at elevated pressure at temperatures far above 100°C.

The treatment time in the invention can be relatively short, for example less than two hours, so that it is differentiated by one order of magnitude from the treatment time in the production of cellulose. The purpose is the removal, of only fractions of the wood which are unwanted with respect to the use as filter aid, i.e. have an effect in terms of flavor, odor and/or color in the filtrate. This is not in this case primarily lignin, but compounds such as essential oils, terpeneoils and terpenoids, tannic acids, fats and waxes, phenolic substances (lignans, phenylpropanes, coumarin) stilbenes, flavonoids and the like, which make up an amount of from approximately 4 to 5 percent by weight of the dry wood. It has been found that these compounds can be, by means of a treatment with dilute alkali solutions acids [sic] even at room temperatures under atmospheric pressure, extracted from the wood or else made inactive to the extent that the treated wood particles are sufficiently neutral sensorially for the practical use as filter aid. It is not the case that during a rigorous analysis no residues of the unwanted type can be observed any longer, but that, for example, a medium filtered using the filter aid does not permit, during sensory testing, any wood flavor or wood aroma or any brown discoloration to be recognized. The treatment can be brief relative to the treatment durations of cellulose production.

An important feature in the treatment of the particles is in addition, that the treatment can also take place at temperatures below 100°C and simultaneously at atmospheric pressure, which substantially simplifies the plant required for producing the filter aid.

The filter aid of the invention can be prepared particularly economically. The costs may be in the same order of magnitude as the costs of kieselguhr, but only about one third of the costs for cellulose powder.

It also appears to be the case that the particles treated according to the invention have on the surface an additionally roughened or fissured structure which advantageously affects the filter properties.

Underlying the ~~[preamble of claim 1]~~ **claimed invention** is French Patent 385 035 which has, as its subject matter, wood fibers used for filter purposes which are treated, to avoid rotting, with a permanganate solution with or without addition of acid.

From JP-A 73 28 353, DE 41 10 252 C1 and EP 747 104 A2 can be taken the use of cellulose fibers that is not alkali-solution-treated wood particles mixed with other components as filter aid.

From FR 534 288 there arises a filter aid for wines, which aid consists of wood wool which is thoroughly washed and treated with 1% strength tartaric acid.

According to ~~[claim 4]~~ **the invention** the filter aid can comprise essentially only wood particles of one and the same type, size and pretreatment, that is can essentially be uniformly composed.

However, it is also possible according to ~~[claim 5]~~ **the invention** for the filter aid to comprise at least two particle fractions comminuted according to different processes, in order to be able to set the filtration properties in accordance with the requirements.

From the same aspect, the filter aid can comprise at least two particle fractions comminuted to different dimensions ~~[(claim 6)]~~ and/or at least two particles produced from different starting materials~~[(claim 7)]~~.

The filter aid can also comprise other fractions which do not affect the filtration properties~~[(claim 8)]~~.

It can also be a mixture with other filter-active constituents, i.e. not consisting of plant fibers~~[(claim 9)]~~, also with mineral constituents~~[(claim 10)]~~, namely

with kieselguhr~~[(claim 11)]~~, which would have the effect of reducing the kieselguhr content and the associated problems mentioned at the outset.

However, suitable additional constituents are also other mineral filter aids, in particular perlite~~[(claim 12)]~~. ~~{~~

~~}~~According to ~~{claim 13}~~ **the invention**, the largest mean particle diameter of the ready-to-use filter aid shall be less than 3.0 mm. ~~{~~

~~}~~In contrast, in the case of fibrous particles, the mean fiber diameter shall be less than 1.0 mm~~[(claim 14)]~~.

Since the particles are produced by grinding, they do not have an exact size, but a size distribution for instance according to a Gaussian curve. The position of the maximum of this curve may be taken to mean here the highest particle dimension.

The filter aid of the invention can be used to form prefloat filter layers in the same manner as was previously the case with mineral filter aids.

The invention also extends to a process ~~{according to claim 15}~~ for preparing the filter aid in which the particles are digested by the treatment liquid in the course of a period of action.

A suitable temperature range in the treatment of the particles is the range of room temperature, which, although it requires no heating energy consumption, does require longer treatment times~~[(claim 16)]~~. ~~{~~

~~}~~A further practicable region with shorter treatment times is 50-100°C~~[(claim 17)]~~.

According to ~~{claim 18}~~ **the invention**, atmospheric pressure in the temperature range of 70 to 90°C can be employed, which is a temperature markedly increased with respect to room temperature, but is below the boiling point and eliminates the use of pressure vessels. This leads to a usable filter aid with a minimum of equipment and energy consumption.

"Dilute alkali solution" shall mean an aqueous solution having a content of from 2 to 10% by weight of the dry alkali, based on the solids content~~[(claim 19)]~~. ~~{~~

[In the preferred embodiment of the invention, sodium hydroxide solution is used[(claim 20)].

The period of action depends apart from the pressure and temperature, on the solution capacity of the dilute alkali solution for the unwanted constituents. In the case of dilute alkali solution, it is also the case that it is not periods of action in the seconds region which come into question, but those which are short in comparison with the periods of action of many hours to days necessary in cellulose production. The period of action is partly dependent on the particle size.

It is, moreover, of a size determined by the fact that precisely only the sensory-critical substances are to be removed from the particles, in particular the wood particles. The latter purpose is achieved when at most 10% by weight on an absolutely dry basis of the wood constituents are removed[(claim 21)], whereas the production of cellulose relates to the liberation of generally more than 30% of the wood constituents.

The period of action can be, with an alkali solution treatment, in particular between 5 and 120 min. [(claim 22).

[The consistency, i.e. the proportion by weight of the particles in the dilute alkali solution, can be from 5 to 25% in the treatment[(claim 23)]. [

[The particles, after the period of action, can be washed and dried[(claim 24)]. [

[The particle size (maximum of the particle size distribution) can be up to 10 mm, preferably 01 to 1.0 mm, during the treatment[(claim 25)].

Since a grinding in the wet phase changes the particle shape, a possibility is opened up in this manner of setting the water value[(claim 26)]. [

[In individual cases it is possible, without relinquishing the lack of sensory hazard to further comminute the particles after the alkali solution treatment and the drying, simultaneously with the drying or after the drying[(claim 27)]. [

[To obtain clear conditions with respect to the filter properties, it is advisable [according to claim 28] to classify the particles after the alkali solution treatment and the drying.

The invention is also embodied in the use of finely divided wood particles which have been subjected to a treatment with a dilute lute [sic] at a temperature below 100°C and at atmospheric pressure, which treatment removes the sensorially active substances from the wood particles, as filter aid ~~{(claim 29)}~~, in particular when the particles have been treated ~~{according to the process of claims 15 to 28 (claim 30)}~~. ~~{~~

~~}~~A suitable use is in particular in beverage filtration, in particular beer filtration~~{(claim 31)}~~.

Other fields of application of the invention are food filtration~~{(claim 32)}~~, for example sugar solutions, edible oil, fat, gelatin, citric acid, alginate etc., filtration in the chemical sector~~{(claim 33)}~~, for example chloralkali, in the sector of the cleaning of auxiliary liquids in metalworking~~{(claim 34)}~~, for example cutting fluids, rolling oils, polishing oils, etc., and in the pharmaceutical and cosmetics sector~~{(claim 35).~~

~~}~~.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

To study the efficacy of the novel treatment of the plant fiber particles, untreated plant fiber particles (Lignocel C 120) were compared with plant fiber particles treated according to the invention (Sample No. 1; Sample No. 2; Sample No. 3). The Samples No. 1 to No. 3 were treated as follows:

Sample 1: To produce the treated plant fiber particles, 330 g of wood fiber flour (particle range: 70-150 Πm), 3700 ml of water and 15.8 g of solid sodium hydroxide were digested (reacted) in a mixing and treatment reactor at from 20°C to 25°C without additional heating and without stirring. The solids content was below 10% by weight, the retention time was at least 16 hours, the pH of the aqueous alkali solution was below 11.3 after 16 hours.

The sodium hydroxide solution was filtered off by vacuum via a plastic filter, the predried wet cake was slurried in hot water (70°C), so that a solids content below 15% by weight was achieved. A final pH of from 3.0 to 7.0 was set using dilute hydrochloric acid and the solution was filtered off under vacuum via a plastic filter. The subsequent rinsing was performed at least twice each time with 200 to 500 ml of water at 70°C.

Sample 2 was treated with hot alkali solution and rinsed cold. In a mixing and treatment reactor at temperatures above 50°C and with stirring 330 g of wood fiber flour (particle range: 70 - 150 µm), 3700 ml of water and less than 12 g solid sodium hydroxide were digested (reacted). The solids content was below 10% by weight; the retention time was at least 20 minutes; the pH value of the aqueous alkali solution was below 10.8 at the end of the experiment. The sodium hydroxide solution was filtered off under vacuum via a plastic filter; the predried wet cake was slurried in hot water (70°C) so that a solids content below 15% by weight was achieved. A final pH ranging from 3.0 to 7.0 was set using dilute hydrochloric acid; and the solution was filtered off by vacuum via a plastic filter. The subsequent rinsing was performed at least twice each time with 200 to 500 ml of cold water at 20°C.

Sample 3 was produced at a pilot plant. The solids content was comparable with the laboratory batches. It was washed three times in cold water.

To determine the yield a thin layer of the resulting wet cake ranging from 5 to 10 mm was spread on a sheet and dried.

The degree of whiteness and the density was determined with this material.

The yield (on an absolutely dry basis) was at least 97% by weight, i.e. at most 3% by weight of the constituents of the wood fiber flour that was employed was released during the alkali solution treatment.

The sensorial test was conducted in an aqueous suspension, in which 1 g of product was suspended in 150 ml water at 100°C. With this suspension the smell and taste were tested.

To get an impression of what is still present in the untreated wood particle material (Lignocel C 120), on the one hand, and the wood particle material (sample nos. 1-3), subjected to the alkali solution treatment, the materials were subjected to an extraction in a Soxleth apparatus. The amount of the still extractive constituents present in the materials is a measure for the suitability of the material as a filter aid for sensorially challenging filtrations.

During the extraction operation in the Soxleth apparatus 5 g of the product dried to a moisture content below 10% by weight were extracted with 250 ml ethanol / water (1:1) for 5 hours; and the extract content was determined gravimetricly.

With the material dried to a moisture content below 10% by weight, a test filtration was subsequently conducted according to a procedure specified by the Schenk company at 20°C, in which procedure the wet cake height, the Darcy value, the fluming behavior, and the water value are determined.

The results of the test are compiled in the attached table.

The sensorial area was evaluated with numbers, where 0 denotes good, 10 denotes poor.

The table shows that the untreated material with respect to odor exhibits a value 8, which is obviously worse than the value of the treated samples nos. 1-3.

The same applies to the flavor, which was virtually impossible to evaluate with the untreated product Lignocel C 120.

An important point is the amount of extract. In the untreated product Lignocel C 120 3.37% could still be extracted, whereas the equivalent values of treated products were 1.0%. This means that the relatively mild alkali solution treatment had already released a noticeable amount of the extractable constituents, a feature that may be a disturbing factor in the use of the product as a filter aid.

The alkali solution treatment of the product and the subsequent washing operations make it possible to affect somewhat the water value, which is a measure for the permeability of the filter aid. The water value is determined with a laboratory pressure filter (diameter 50 mm) and an elevated water tank with level control. Between the level of the water in the elevated water tank and the filter bottom a difference of 2 m must be maintained.

The laboratory filter is provided and sealed with a moistened permeable layer of cellulose (Schenk D layer with the screen side downward). Then 25 g of filter aid are slurried in 200 to 300 ml pure water and completely transferred into the lab filter. The lab filter is attached to the elevated water tank and purged. After one

minute 500 ml water are removed via a filter and then the time for the next 100 ml filtrate stopped. The water value follows from the stopped time as follows:

$$\text{water value} = \frac{480}{\text{time in minutes}}$$

If the result is a water value of less than 150, the determination is done as above, but with the use of only 4 g of filter aid. Then the result is:

$$\text{water value} = \frac{76.8}{\text{time in minutes}}$$

Hence the shorter the time, required for a specific volume of water to flow through the filter layer, the higher is the water value.

Table

Product	Moisture content % by weight	Odor	Flavor	Turbidity	Color	Extract Whiteness %	Bulk ~~[density]~~ Density g/dm^3	Wet cake height mm/25g	Darcy Value	Incoming flow behavior	Water value min^1/25 g
Reference Lignocel C120	9.0	8	10 (bitter)	8 (yellow)	3.37	58.4	128	78	5.3	good	770
Sample No. 1	3.5	1	2-3 (mild)	1-2 (Colorless	0.93	34.2	Not determined	83	8.0	good	1098
Sample No. 2	5.3	1	2-3 (mild)	2 (Colorless	1.04	34.1	125	82	7.3	good	1010
Sample No. 3	7.5	5	6 (neutral)	2-3 (almost colorless)	0.98	36.8	131	79	7.8	good	1125

~~[WE CLAIM]~~ WHAT IS CLAIMED IS:

1. Filter aid which comprises finely divided wood particles which have been subjected to a chemical liquid treatment, characterized in that the particles have been subjected to a treatment with a dilute alkali solution at a temperature below 100°C and at atmospheric pressure, which removes the sensorially active substances from the wood particles.

2. Filter aids according to claim 1, characterized in that the particles comprise wood fibers.

3. Filter aids according to claim 1, characterized in that the particles comprise wood comminution residues.

4. Filter aid according to one of claim 1, characterized in that it essentially comprises only wood particles of one and the same type, size distribution and pretreatment.

5. Filter aid according to one of claim 1, characterized in that it comprises at least two fractions of particles comminuted by different processes.

6. Filter aid according to one of claim 1, characterized in that it comprises at least two fractions of particles comminuted to different dimensions.

7. Filter aid according to one of claim 1, characterized in that it comprises fractions of particles produced from at least two different starting materials.

8. Filter aid according to one of claim 1, characterized in that it comprises other organic or inorganic fractions which do not affect the filtration properties.

9. Filter aid according to one of claim 1, characterized in that it comprises other filter-active fractions.

10. Filter aid according to one of claim 1, characterized in that it comprises other mineral fractions.

11. Filter aid according to one of claim 1, characterized in that it comprises kieselguhr.

12. Filter aid according to one of claim 1, characterized in that it comprises perlite.

13. Filter aid according to one of claim 1, characterized in that the mean particle dimension of the ready-to-use filter aid is below 3.0 mm.

14. Filter aid according to one of claim 1, characterized in that the mean fiber diameter is below 1.0 mm in the case of fibrous particles.

15. Process for producing the filter aid according to claim 1, characterized in that the particles are digested with the dilute alkali solution during a period of action.

16. Process according to claim 15, characterized in that the temperature of the dilute alkali solution during the treatment is in the range of room temperature.

17. Process according to claim 15, characterized in that the temperature of the dilute alkali solution during treatment is 50-100°C.

18. Process according to claim 15, characterized in that the temperature of the dilute alkali solution during the treatment is from 70 to 90°C.

19. Process according to claim 15, characterized in that concentration of the dilute alkali solution is from 2 to 10% by weight, based on the solids content.

20. Process according to claim 15, characterized in that the alkali solution used is sodium hydroxide solution.

21. Process according to claim 15, characterized in that the period of action is of a duration such that at most 10% by weight on an absolutely dry basis of the wood constituents are removed.

22. Process according to claim 15, characterized in that the period of action is from 5 to 120 min.

23. Process according to claim 15, characterized in that the consistency during the treatment is from 5 to 25%.

24. Process according to claim 15, characterized in that the particles are washed and dried after the period of action.

25. Process according to claim 15, characterized in that the particle size during the treatment is up to 10 mm, preferably from 0.1 to 1.0 mm.

26. Process according to claim 15, characterized in that the water value is set by influencing the grinding in the wet phase (refiner).

27. Process according to claim 15, characterized in that the particles are further comminuted after the treatment and before the drying, simultaneously with the drying or after the drying.

28. Process according to claim 15, characterized in that the particles are classified after the treatment and the drying.

29. The use of finely divided wood particles which have been subjected to a treatment with a dilute alkali solution at a temperature below 100°C and at atmospheric pressure, which treatment removes the sensorially active substances from the wood particles, as filter aid.

30. The use of finely divided wood particles which have been treated according to claim 15 as filter aid.

31. The use according to claim 29 in beverage filtration, in particular beer filtration.

32. The use according to claim 29 in food filtration.

33. The use according to claim 29 in the sector of the cleaning of liquids in the chemicals industry.

34. The use according to claim 29 in the sector of the cleaning of auxiliary liquids in metalworking.

35. The use according to claim 29 in the sector of pharmaceuticals and cosmetics.

ABSTRACT OF THE DISCLOSURE

The filter aid comprises finely divided plant fibers, which for a period of action have been subjected to a liquid treatment, which removes the sensorially active substances from the plant fibers.

FIRST STAGE MARK-UP

TRANSLATION AND SUBSTITUTE PAGES ARE INTERLEAVED AND REDUNDANT MATTER IS MARKED FOR DELETION

Filter Aid

The invention relates to a filter aid, as disclosed in the preamble of claim 1, a proceess for its production and its application.

Cellulose-based filter aids have been known for a long time ("Ullmann's Encyklopädie der technischen Chemie", 3rd edition (1951), first volume, page 492, key word "felted layers" and page 493, key word "filter aids"). Cellulose is produced in a multi-step chemical process, in which all sensorially active materials are removed from the raw material.

Hence filter aids made of pure cellulose are used wherever the sensorial neutrality of the used filter aid is of great significance. Examples of cellulosic filter aids are EFC (low extract cellulose), fine powder cellulose, fine fibrillated cellulose, cationized powder cellulose, fine MCC (microcrystalline cellulose).

In contrast, filter aids made of untreated woodpulp are produced by mechanical comminution, thus only by physical treatment, and can, thus, release extractives (color, odor, flavor) during filtration. Therefore, the use of wood fiber-based filter aids is usually limited to industrial filtrations, where relatively little demand is placed on the sensory analysis.

Not only for filtration in the food and luxury food sector, but also for many industrial applications, they may not be considered, e.g. for sugar solutions (glucose, dextrose, fructose), molasses, dye solutions, fats and oils and the like.

The difficult field of beverage filtration demands, on the one hand, complete sensorial neutrality of the used filter aid; on the other hand, the number of commonly used filter aids are limited for economic reasons, since the maximum expense for the filter aid is fixed by the price of the mineral filter aids dominating this market.

Usually beer filtration take place in two steps. The first step usually involves a coarse filtration, during which operation the liquid usually passes through a precoated layer of a filter aid. This step is frequently followed by a fine filtration (membrane, kieselguhr, etc.).

The standard filter aid for the precoat-type filter in the beverage, especially beer, sector, is kieselguhr. A high percentage of the world beer production is clarified by kieselguhr filtration. Currently it exceeds more than 1.1 billion hl beer.

The total demand for filter aids is worldwide ca. 750,000 tons per year, where inorganic materials, like, for example, kieselguhr, perlite or bentonite, constitute by far the largest share of this amount. Of this total amount about 250,000 tons to 300,000 tons per year are consumed worldwide by the beverage industry, largely by the breweries, but also by producers of wine and fruit juices.

The amount of filter aids, which are based on organic raw materials that can regrow again (cellulose, woodpulp, etc.), is to date only ca. 20,000 tons per year, even though their use offers a plurality of advantages over inorganic filter aids.

Thus organic filter aids are natural materials, whose quality fluctuates only over a narrow range and whose occurrence can be renewed at regular intervals. In addition the use of organic filter aids presents neither a health risk nor harmful effects for the environment and nature. Pumps and conveying elements of the filtration system are protected as much as possible owing to the non-abrasive property. Finally the consumed filter cakes can be easily disposed, for example, through land management, composting or animal fodder.

Of course, organic filter aids are in part many times more expensive than kieselguhr or they exhibit filtration properties that do not completely match those of kieselguhr.

For this reason organic filter aids have not been able to prevail to date against kieselguhr or are used in any case together with kieselguhr (report by J. Speckner "Cellulose as Filter Aids" in the journal ("Brauwelt", vol. 124 (1984), issue 46, pages 2058 to 2066, in particular page 2062, left column top).

However, kieselguhr has become increasingly a problem. As a natural mineral material its occurrence is limited. Thus in the case of kieselguhr one must resort more and more to low grade qualities in order to meet the high demand of industry. The result is, however, a rising cost for the cleaning and processing of kieselguhr, which could in the long run have a negative impact on its economic situation.

An even greater impact presents, however, the fact that the users' attitude toward kieselguhr is becoming more critical.

This reservation stems from the problems posed to the lungs due to many natural mineral materials and hence kieselguhr, a feature that must be taken very seriously from the point of view of occupational medicine. In 1988 the World Health Organization (WHO) categorized kieselguhr as a carcinogenic substance following a series of animal experiments. For handling there are strict regulations that are being asserted and observed more and more in Germany.

Another factor is that the disposal of kieselguhr is becoming increasingly more critical in industrial countries. A classification as hazardous waste makes landfilling considerably more difficult. With the introduction of the new technical rules on municipal waste, the disposal situation for kieselguhr is becoming more restrictive. In many cases disposal of kieselguhr used as filter aid already gives rise to costs of approximately DM 600.00 per t of kieselguhr, when used in beer filtration, or 1,500.00 per t of kieselguhr when used in the industrial filtration of problematic substances.

Starting from these urgent problems in particular in the beer filtration sector, the invention is based on the problem of developing a filter aid that is effective and can be provided economically.

This problem is solved by the invention described in claim 1.

Sensorially active substances, thus active with respect to color, odor and / or flavor, are to be removed from the filter aid to a sufficient extent prior to use as a filter aid so that none of these substances can pass into the filtrate to a significant extent and impair its sensorial properties. The particles are neutralized sufficiently to a certain extent sensorially in order to be able to serve as a filter aid. In so doing, it is essential that the treatment is carried out only to the extent that it is necessary for this purpose. The use of energy and chemicals remains in a justifiable range so that the product can compete economically with kieselguhr. The action is, therefore, not as intensive as in the case of the production of cellulose from wood fibers. Surprisingly it has been found that by means of a liquid treatment a sufficient neutralization of the particles with respect to sensorial aspects can be achieved without the need for simultaneously high pressures and temperatures to be employed, without the need for large amounts of aggressive chemicals and treatment periods of many hours to days. In this respect the invention opens an expanded area of application for plant fiber materials without the expense required for the production of cellulose.

Although the starting point and preferred area of application for the invention is beer and beverage filtration, which concerns the provision of a kieselguhr substitute material, the invention is not restricted to this area of application.

In a preferred embodiment of the inventive thought the particles comprise wood particles (claim 2), for example, wood fibers (claim 3) or, in particular, wood comminution residues (claim 4), hence for example sawdust, sanding dust, wood shavings, wood chips, cutting waste, chipped wood and the like.

However, cotton, straw, hemp, flax, bast, grasses are also suitable starting material, similarly cellulose-containing secondary raw materials, like old paper and paper waste.

In the preferred embodiment of the invention the particles have been treated with a dilute alkali solution (claim 5). However, treatment with diluted acid (claim 6), with an organic or inorganic solvent (claim 7) or also only with water (claim 8) is not ruled out, where according to the

~~conventional rules longer action periods and higher temperature must compensate for the lower~~ solubilizing power.

The filter aid particles produced from wood particles are actually to retain their wood character, i.e. the lignin shall not have been virtually quantitatively extracted from the raw material wood, as occurs in cellulose production in the sulfite or sulfate process by treatment for many hours at raised pressure at temperatures far above 100°C.

The treatment time in the invention can be relatively short, especially for the alkali solution treatment, for example less than two hours, so that it is differentiated by almost one order of magnitude from the treatment time in the production of cellulose. The goal is the removal of only fractions of the wood that are unwanted with respect to the application purpose as a filter aid, i.e. have an effect in terms of flavor, odor and/or color in the filtrate. In this case it is not primarily lignin, but compounds, such as essential oils, terpeneoils and terpenoids, tannic acids, fats and waxes, phenolic substances (lignans, phenylpropanes, coumarin) stilbenes, flavonoids and the like, which constitute an amount ranging from approximately 4 to 5 percent by weight of the dry wood. It has been found that these compounds can be, by means of a treatment with dilute alkali solutions or acids even at ambient temperatures under atmospheric pressure, extracted from the wood or else made inactive to the extent that the treated wood particles are sufficiently neutral sensorially for practical use as a filter aid. It is irrelevant whether during a rigorous analysis no residues of the unwanted type can be observed any longer, but rather, for example, a medium filtered using the filter aid does not permit, during sensorial testing, any wood flavor or wood aroma or any brown discoloration to be recognized.

Precisely with the use of wood particles as the starting product can the filter aid of the invention be provided especially economically. The cost ought to be in the same order of magnitude as the cost for kieselguhr, but only at about one-third the cost for cellulose powder.

It also seems to the case that the particles treated according to the invention also exhibit a roughened or fissured surface structure that has an advantageous effect on the filter properties.

The treatment can be brief relative to the treatment durations of cellulose production.

The grinding determines largely the filtration properties. With fine grinding the permeability of the filter layer is generally lower. By means of grinding (micronization, fibrillation), the particle shape is also affected, a feature that in turn changes the water value, which will be explained below, as a measure for the permeability of the filter layer. In the case of fibrous cellulose products they can be fibrillated to a greater or lesser extent. The grinding can also be performed in a plurality of steps, during which procedure a first grinding for the production of the particles is followed by a further ~~grinding after the treatment and before or after the drying.~~

~~According to claim 9, the filter aid can comprise essentially only wood particles of one and the~~ same type, size and pretreatment, thus can be essentially uniformly composed.

However, it is also possible according to claim 10 for the filter aid to comprise at least two particle fractions comminuted according to different processes, in order to be able to set the filtration properties in accordance with the requirements.

From the same aspect, the filter aid can comprise at least two particle fractions comminuted to different dimensions (claim 11) and/or at least two particles produced from different starting materials (plant fibers) (claim 12).

The filter aid can also comprise other fractions that do not affect the filtration properties (claim 13).

It can also be a mixture with other filter-active constituents, i.e. not consisting of plant fibers (claim 14), also with mineral constituents (claim 15), namely with kieselguhr (claim 16), which would have the effect of reducing the kieselguhr content and the associated problems mentioned at the outset.

However, suitable additional constituents are also other mineral filter aids, in particular perlite (claim 17).

According to claim 18, the largest mean particle diameter of the ready-to-use filter aid shall be less than 3.0 mm.

In contrast, in the case of fibrous particles the mean fiber diameter shall be less than 1.0 mm.

Since the particles are produced by grinding, they do not have an exact size, but rather a size distribution for instance according to a Gaussian curve. The position of the maximum on this curve may be taken to mean here the largest particle dimension.

The filter aid of the invention can be used to form precoated filter layers in the same manner as was previously the case with mineral filter aids.

The invention also extends to a process according to claim 20 for producing the filter aid, in which the particles are digested by the treatment solution in the course of a period of action.

A suitable temperature range during the treatment of the particles is the range of ambient temperature, which, although it does not require any consumption of heating energy, does require longer treatment times (claim 21).

~~Another practicable range with shorter treatment times is 50-130°C (claim 22).~~

~~Another important feature of the process during the treatment of the particles is, however, that the~~ treatment can also take place at temperatures below 100°C and simultaneously at atmospheric pressure (claim 23), a feature that significantly simplifies the system required to produce the filter aid.

In a preferred method according to claim 24 a dilute alkali solution is used as the treatment medium.

According to claim 25, atmospheric pressure in the temperature range of 70 to 90°C can be employed, which is a temperature markedly increased with respect to room temperature, but is below the boiling point and eliminates the use of pressure vessels. This leads to a usable filter aid with a minimum of equipment and energy consumption.

"Dilute alkali solution" shall mean an aqueous solution having a content ranging from 2 to 10% by weight of the dry alkali, based on the solids content (claim 26).

In the preferred embodiment of the invention, a sodium hydroxide solution is used (claim 27).

The period of action depends, apart from the pressure and temperature, on the solubility power of the treatment solution for the unwanted constituents and in the case of water as the treatment solution it will be relatively the longest. In the case of a dilute alkali solution as the treatment solution, periods in the seconds range are out of the question, but rather those that are short in comparison with the periods of action of many hours to days necessary in cellulose production. The period of action is partly dependent on the particle size.

It is, moreover, of a size determined by the fact that precisely only the sensory-critical substances are to be removed from the particles, in particular the wood particles. The latter purpose is achieved when at most 10% by weight on an absolutely dry basis of the wood constituents are removed (claim 28), whereas the production of cellulose relates to the release of generally more than 30% of the wood constituents.

In the case of an alkali solution treatment the period of action can range in particular from 5 to 120 min. (claim 29).

The density, i.e. the percentage by weight of the particles in the dilute alkali solution, can range from 5 to 25% during the treatment (claim 30).

After the period of action the particles can be washed and dried (claim 31).

The particle size (maximum of the particle size distribution) can be up to 10 mm, preferably 0.1 to ~~1.0 mm, during the treatment (claim 32).~~

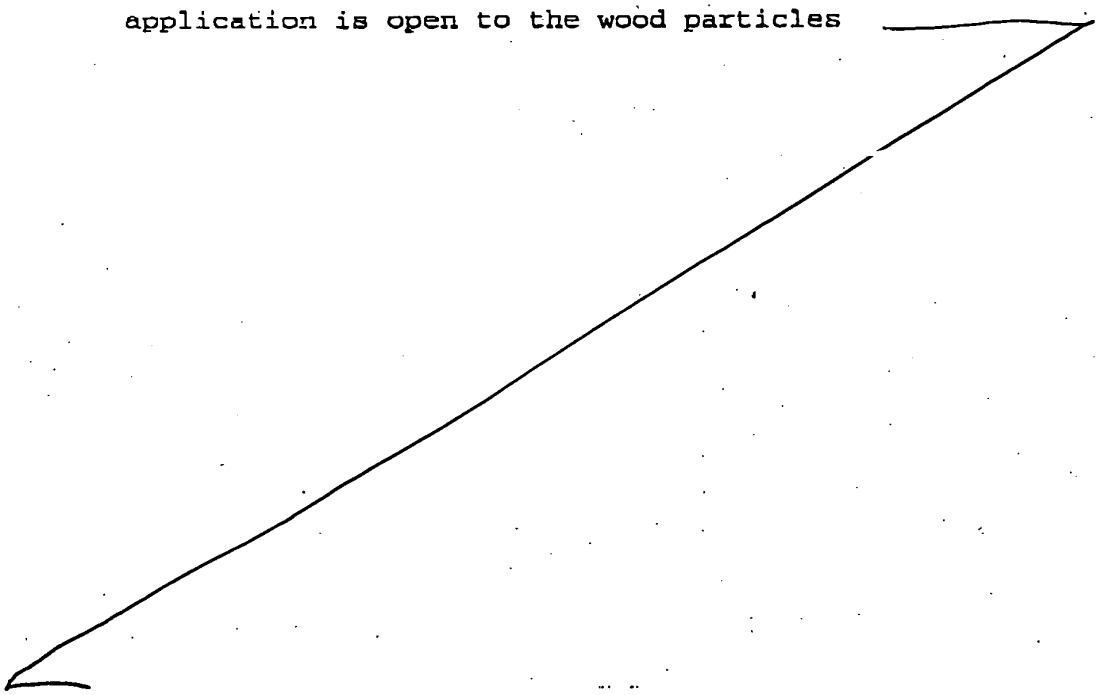
kieselguhr is becoming increasingly more critical in industrial countries. A classification as hazardous waste makes landfilling considerably more difficult. With the introduction of the new technical rules on municipal waste, the disposal situation for kieselguhr is becoming more restrictive. In many cases, disposal of kieselguhr used as filter aid already gives rise to costs of approximately DM 600,000 per t of kieselguhr, if this was used in beer filtration, or 1500.00 per t of kieselguhr if this was used in the industrial filtration of problematic substances.

Starting from these urgent problems in particular in the beer filtration sector, the object underlying the invention is to develop a filter aid which is effective and can be provided economically.

This object is achieved by the invention described in claim 1.

Substances which are sensorially active, that is active with respect to color, odor and/or flavor, are to be removed from the filter aid to a sufficient extent prior to use as filter aid, so that none of these substances can transfer into the filtrate to a significant extent and impair its sensory properties. The particles are neutralized sufficiently to a certain extent sensorially in order to be able to serve as filter aid. It is essential here that the treatment is

only carried out to the extent necessary for this purpose. The use of energy and chemicals remains in a justifiable range, so that the product can compete economically with kieselguhr. The action is therefore not so intensive as is the case in the production of cellulose from wood fibers. Surprisingly, it has been found that by means of a liquid treatment a sufficient neutralization of the particles with respect to sensory aspects can be achieved without the need for simultaneously high pressures and temperatures to be employed, large amounts of aggressive chemicals and treatment times of many hours to days. By means of the invention, to a certain extent an expanded area of application is open to the wood particles



, without this needing expenditure as in the case of cellulose production.

Although the starting point and preferred area of application for the invention is beer and beverage filtration, which concerns the creation of a kieselguhr substitute material, the invention is not restricted to this area of application.

Although DE 23 51 125 A1 discloses using, for the extraction of metals or metal ions from solutions, an adsorbent which is obtained by bringing, inter alia, wood sawdust into contact with a solution of a hydroxide of an alkali metal or alkaline earth metal, this is an adsorption, i.e. an accumulation of dissolved particles on the surface of the adsorbent, not a filtration, i.e. a separation from a suspension.

In the case of DE 41 10 252 C1, for prefloating a filter cake, a mixture of filter aids of differing morphological and physical properties is used which comprise at least one component which increases the density of the filter cake and is made of high-density, chemically resistant metal and/or metal oxide and/or carbon particles of fibrous and/or granular structure and a further component of plastic fibers and/or cellulose fibers having a fiber length of from 1000 to 5000 µm and a fiber thickness of from 0.5 to 100 µm.

The wood particles of the filter aid according

to the invention comprise, for example, wood fibers (claim 2) or, in particular, wood comminution residues (claim 3), that is for example sawdust, sanding dust, wood shavings, wood chips, cutting waste, chipped wood and the like.

The grinding performed in the production of the wood particles in the invention substantially determines the filtration properties. With fine grinding, the permeability of the filter layer is generally lower. By means of the grinding (micronization, fibrillation), in addition the particle shape is affected which in turn changes the water value, which is still to be explained, as a measure of the permeability of the filter layer. In the case of fibrous cellulose products these can be fibrilated to a greater or lesser extent. The grinding can also be performed in a plurality of steps in which a first grinding for the production of the particles is followed by a further

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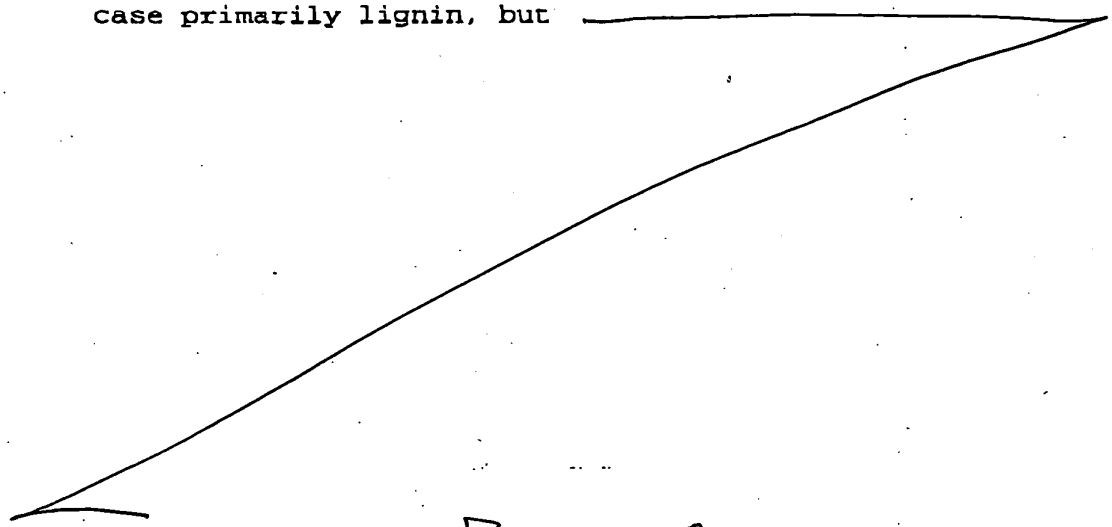
grinding after the treatment and before or after the drying.

The filter aid particles produced from wood particles are still actually to have wood character, i.e. the lignin shall not have been virtually quantitatively extracted from the raw material wood, as occurs in cellulose production in the sulfite or sulfate process by treatment for many hours at elevated pressure at temperatures far above 100°C.

The treatment time in the invention can be relatively short, for example less than two hours, so that it is differentiated by one order of magnitude from the treatment time in the production of cellulose. The purpose is the removal of only fractions of the wood which are unwanted with respect to the use as filter aid, i.e. have an effect in terms of flavor, odor and/or color in the filtrate. This is not in this case primarily lignin, but



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compounds such as essential oils, terpen oils and terpenoids, tannic acids, fats and waxes, phenolic substances (lignans, phenylpropanes, coumarin) stilbenes, flavonoids and the like, which make up an amount of from approximately 4 to 5 percent by weight of the dry wood. It has been found that these compounds can be, by means of a treatment with dilute alkali solutions acids [sic] even at room temperatures under atmospheric pressure, extracted from the wood or else made inactive to the extent that the treated wood particles are sufficiently neutral sensorially for the practical use as filter aid. It is not the case that during a rigorous analysis no residues of the unwanted type can be observed any longer, but that, for example, a medium filtered using the filter aid does not permit, during sensory testing, any wood flavor or wood aroma or any brown discoloration to be recognized. The treatment can be brief relative to the treatment durations of cellulose production.

An important feature in the treatment of the particles is, in addition, that the treatment can also take place at temperatures below 100°C and simultaneously at atmospheric pressure, which substantially simplifies the plant required for producing the filter aid.

The filter aid of the invention can be prepared

particularly economically. The costs may be in the same order of magnitude as the costs of kieselguhr, but only about one third of the costs for cellulose powder.

It also appears to be the case that the particles treated according to the invention have on the surface an additionally roughened or fissured structure which advantageously affects the filter properties.

Underlying the preamble of claim 1 is French Patent 385 035 which has, as its subject matter, wood fibers used for filter purposes which are treated, to avoid rotting, with a permanganate solution with or without addition of acid.

From JP-A 73 28 353, DE 41 10 252 C1 and EP 747 104 A2 can be taken the use of cellulose fibers, that is not alkali-solution-treated wood particles

, mixed with other components as filter aid.

From FR 534 288 there arises a filter aid for wines, which aid consists of wood wool which is thoroughly washed and treated with 1% strength tartaric acid.

According to claim 4 the filter aid can comprise essentially only wood particles of one and the same type, size and pretreatment, that is can essentially be uniformly composed.

However, it is also possible according to claim 5 for the filter aid to comprise at least two particle fractions comminuted according to different processes, in order to be able to set the filtration properties in accordance with the requirements.

From the same aspect, the filter aid can comprise at least two particle fractions comminuted to different dimensions (claim 6) and/or at least two particles produced from different starting materials (claim 7).

The filter aid can also comprise other fractions which do not affect the filtration properties (claim 8).

It can also be a mixture with other filter-active constituents, i.e. not consisting of plant fibers (claim 9), also with mineral constituents (claim 10), namely with kieselguhr (claim 11), which would

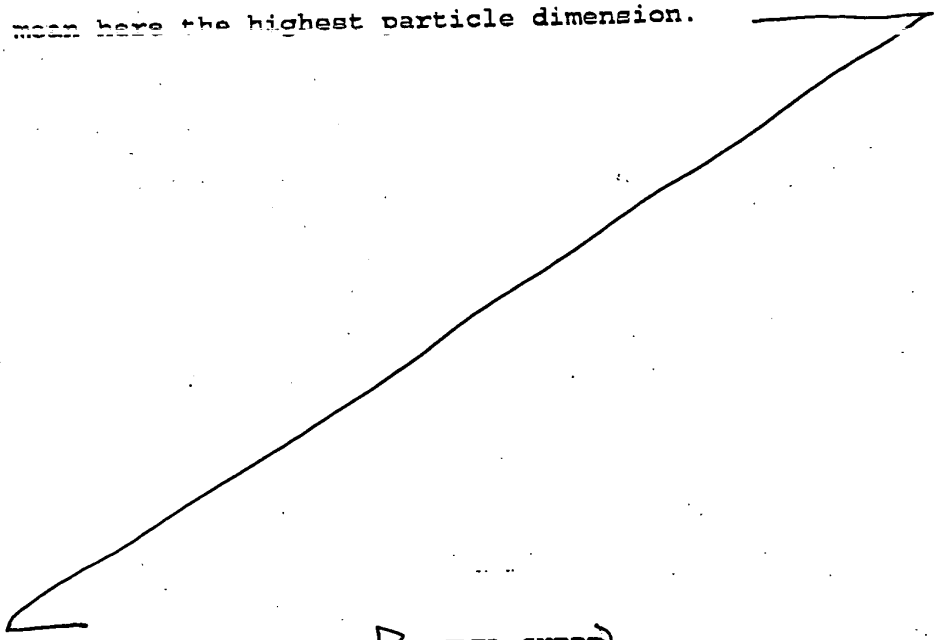
have the effect of reducing the kieselguhr content and the associated problems mentioned at the outset.

However, suitable additional constituents are also other mineral filter aids, in particular perlite (claim 12).

According to claim 13, the largest mean particle diameter of the ready-to-use filter aid shall be less than 3.0 mm.

In contrast, in the case of fibrous particles, the mean fiber diameter shall be less than 1.0 mm (claim 14).

Since the particles are produced by grinding, they do not have an exact size, but a size distribution for instance according to a Gaussian curve. The position of the maximum of this curve may be taken to mean here the highest particle dimension.



The filter aid of the invention can be used to form prefloat filter layers in the same manner as was previously th case with mineral filter aids.

The invention also extends to a process according to claim 15 for preparing the filter aid in which the particles are digested by the treatment liquid in the course of a period of action.

A suitable temperature range in the treatment of the particles is the range of room temperature, which, although it requires no heating energy consumption, does require longer treatment times (claim 16).

A further practicable region with shorter treatment times is 50-100°C (claim 17).

According to claim 18, atmospheric pressure in the temperature range of 70 to 90°C can be employed, which is a temperature markedly increased with respect to room temperature, but is below the boiling point and eliminates the use of pressure vessels. This leads to a usable filter aid with a minimum of equipment and energy consumption.

"Dilute alkali solution" shall mean an aqueous solution having a content of from 2 to 10% by weight of the dry alkali, based on the solids content (claim 19).

In the preferred embodiment of the invention, sodium hydroxide solution is used (claim 20).

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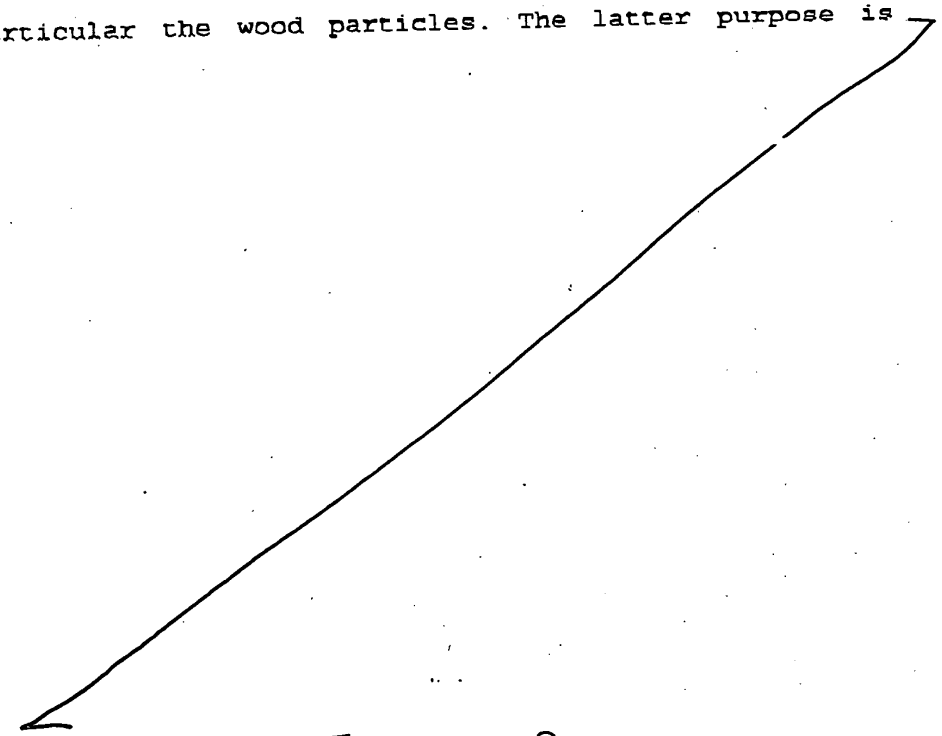
The period of action depends, apart from the pressure and temp rature, on the solution capacity of the dilute alkali solution for the unwanted constituents. In the case of dilute alkali solution, it is also the case that it is not periods of action in the seconds region which come into question, but those which are short in comparison with the periods of action of many hours to days necessary in cellulose production. The period of action is partly dependent on the particle size.

It is, moreover, of a size determined by the fact that precisely only the sensory-critical substances are to be removed from the particles, in particular the wood particles. The latter purpose is



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achieved when at most 10% by weight on an absolutely dry basis of the wood constituents are removed (claim 21), whereas the production f cellulose relates to the liberation of generally more than 30% of the wood constituents.

The period of action can be, with an alkali solution treatment, in particular between 5 and 120 min. (claim 22).

The consistency, i.e. the proportion by weight of the particles in the dilute alkali solution, can be from 5 to 25% in the treatment (claim 23).

The particles, after the period of action, can be washed and dried (claim 24).

The particle size (maximum of the particle size distribution) can be up to 10 mm, preferably 0.1 to 1.0 mm, during the treatment (claim 25).

Since a grinding in the wet phase changes the particle shape, a possibility is opened up in this manner of setting the water value (claim 26).

In individual cases it is possible, without relinquishing the lack of sensory hazard to further comminute the particles after the alkali solution treatment and the drying, simultaneously with the drying or after the drying (claim 27).

To obtain clear conditions with respect to the filter properties, it is advisable according to claim 28 to classify the particles after the alkali solution

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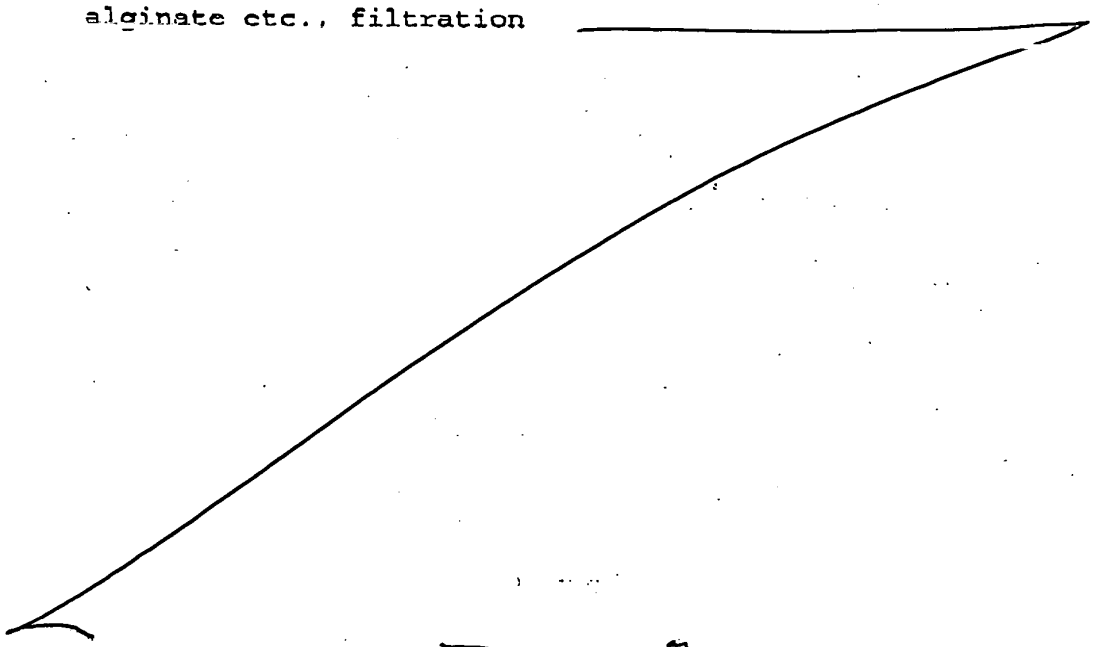
treatment and the drying.

The invention is also embodied in the use of finely divided wood particles which have been subjected to a treatment with a dilute lute [sic] at a temperature below 100°C and at atmospheric pressure, which treatment removes the sensorially active substances from the wood particles, as filter aid (claim 29), in particular when the particles have been treated according to the process of claims 15 to 28 (claim 30).

A suitable use is in particular in beverage filtration, in particular beer filtration (claim 31).

Other fields of application of the invention are food filtration (claim 32), for example sugar solutions, edible oil, fat, gelatin, citric acid, alginate etc., filtration



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in the chemical sector (claim 33), for example chloralkali, in the sector of the cleaning of auxiliary liquids in metalworking (claim 34), for example cutting fluids, rolling oils, polishing oils, etc., and in the pharmaceutical and cosmetics sector (claim 35).

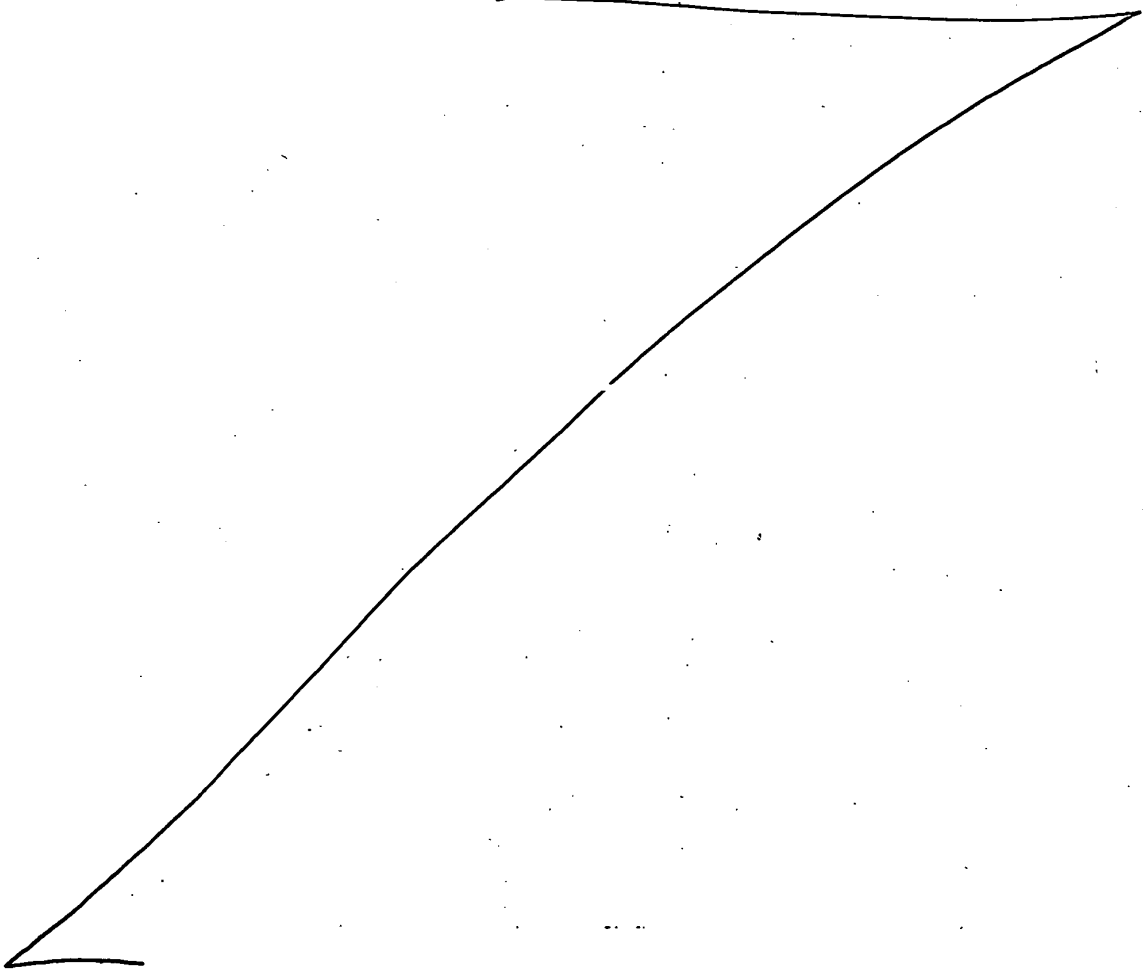
To study the efficacy of the novel treatment of the plant fiber particles, untreated plant fiber particles (Lignocel C 120) were compared with plant fiber particles treated according to the invention (Sample No. 1; Sample No. 2; Sample No. 3). The Samples No. 1 to No. 3 were treated as follows:

Sample 1: To produce the treated plant fiber particles, 330 g of wood fiber flour (particle range: 70 -150 µm), 3700 ml of water and 15.8 g of solid sodium hydroxide were digested (reacted) in a mixing and treatment reactor at from 20°C to 25°C without additional heating and without stirring. The solids content was below 10% by weight, the retention time was at least 16 hours, the pH of the aqueous alkali solution was below 11.3 after 16 hours.

The sodium hydroxide solution was filtered off by vacuum via a plastic filter, the predried wet cake was slurried in hot water (70°C), so that a solids content below 15% by weight was achieved. A final pH of from 3.0 to 7.0 was set using dilute

hydrochloric acid and the solution was filtered off under vacuum via a plastic filter. The subsequent rinsing was performed at least twice each time with 200 to 500 ml of water at 70°C.

Sample 2 was treated with hot alkali solution and rinsed cold. In a mixing and treatment reactor, at temperatures above 50°C and with stirring 330 g of wood fiber flour (particle range: 70 - 150 µm), 3700 ml of water and less than



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~~Since grinding in the wet phase changes the particle shape, a possibility is opened up in this manner~~ of setting the water value (claim 33).

In individual cases it is possible, without having to waive the sensorial neutrality, to further comminute the particles after the alkali solution treatment and the drying process (claim 34).

To obtain clear conditions with respect to the filter properties, it is advisable, according to claim 35, to grade the particles after the alkali solution treatment and the drying process.

The invention is also embodied in the use of finely divided plant particles, which during a period of action have been subjected to a liquid treatment, which removes the sensorially active substances from the plant particles, as a filter aid (claim 36), in particular when the particles have been treated according to the process of claims 20 to 35 (claim 37).

A suitable use is especially in beverage filtration, in particular beer filtration (claim 38).

Other fields of application of the invention are food filtration (claim 39), for example sugar solutions, edible oil, fat, gelatin, citric acid, alginate etc., filtration in the chemical sector (claim 40), for example chloralkali, in the sector of the cleaning of auxiliary liquids in metalworking (claim 41), for example cooling lubricants, rolling oils, polishing oils, etc., and in the pharmaceutical and cosmetics sector (claim 42).

To study the efficacy of the treatment of the plant fiber particles of the invention, untreated plant fiber particles (Lignocel C 120) were compared with plant fiber particles treated according to the invention (Sample No. 1; Sample No. 2; Sample No. 3). The Samples No. 1 to No. 3 were treated as follows:

Sample 1: To produce the treated plant fiber particles, 330 g of wood fiber flour (particle range: 70 -150 mm), 3700 ml of water and 15.8 g of solid sodium hydroxide were digested (reacted) in a mixing and treatment reactor at from 20°C to 25°C without additional heating and without stirring. The solids content was below 10% by weight, the retention time was at least 16 hours, the pH of the aqueous alkali solution was below 11.3 after 16 hours.

The sodium hydroxide solution was filtered off by vacuum via a plastic filter, the predried wet cake was slurried in hot water (70°C) so that a solids content below 15% by weight was achieved. A final pH ranging from 3.0 to 7.0 was set using dilute hydrochloric acid, and the solution was filtered off under vacuum via a plastic filter. The subsequent rinsing was performed at least twice each time with 200 to 500 ml of water at 70°C.

Sample 2 was treated with a hot alkali solution and rinsed cold. In a mixing and treatment reactor at temperatures above 50°C and with stirring 330 g of wood fiber flour (particle range: 70 - 150 ~~mm), 3700 ml of water and less than~~ 12 g solid sodium hydroxide were digested (reacted). The

solids content was below 10% by weight; the retention time was at least 20 minutes; the pH value of the aqueous alkali solution was below 10.8 at the end of the experiment. The sodium hydroxide solution was filtered off under vacuum via a plastic filter; the predried wet cake was slurried in hot water (70°C) so that a solids content below 15% by weight was achieved. A final pH ranging from 3.0 to 7.0 was set using dilute hydrochloric acid; and the solution was filtered off by vacuum via a plastic filter. The subsequent rinsing was performed at least twice each time with 200 to 500 ml of cold water at 20°C.

Sample 3 was produced at a pilot plant. The solids content was comparable with the laboratory batches. It was washed three times in cold water.

To determine the yield a thin layer of the resulting wet cake ranging from 5 to 10 mm was spread on a sheet and dried.

The degree of whiteness and the density was determined with this material.

The yield (on an absolutely dry basis) was at least 97% by weight, i.e. at most 3% by weight of the constituents of the wood fiber flour that was employed was released during the alkali solution treatment.

The sensorial test was conducted in an aqueous suspension, in which 1 g of product was suspended in 150 ml water at 100°C. With this suspension the smell and taste were tested.

To get an impression of what is still present in the untreated wood particle material (Lignocel C 120), on the one hand, and the wood particle material (sample nos. 1-3), subjected to the alkali solution treatment, the materials were subjected to an extraction in a Soxleth apparatus. The amount of the still extractive constituents present in the materials is a measure for the suitability of the material as a filter aid for sensorially challenging filtrations.

During the extraction operation in the Soxleth apparatus 5 g of the product dried to a moisture content below 10% by weight were extracted with 250 ml ethanol / water (1:1) for 5 hours; and the extract content was determined gravimetricly.

With the material dried to a moisture content below 10% by weight, a test filtration was subsequently conducted according to a procedure specified by the Schenk company at 20°C, in which procedure the wet cake height, the Darcy value, the fluming behavior, and the water value are determined.

The results of the test are compiled in the attached table.

The sensorial area was evaluated with numbers, where 0 denotes good, 10 denotes poor.

The table shows that the untreated material with respect to odor exhibits a value 8, which is obviously worse than the value of the treated samples nos. 1 - 3.

The same applies to the flavor, which was virtually impossible to evaluate with the untreated product Lignocel C 120.

An important point is the amount of extract. In the untreated product Lignocel C 120 3.37% could still be extracted, whereas the equivalent values of treated products were 1.0%. This means that the relatively mild alkali solution treatment had already released a noticeable amount of the extractable constituents, a feature that may be a disturbing factor in the use of the product as a filter aid.

The alkali solution treatment of the product and the subsequent washing operations make it possible to affect somewhat the water value, which is a measure for the permeability of the filter aid. The water value is determined with a laboratory pressure filter (diameter 50 mm) and an elevated water tank with level control. Between the level of the water in the elevated water tank and the filter bottom a difference of 2 m must be maintained.

The laboratory filter is provided and sealed with a moistened permeable layer of cellulose (Schenk D layer with the screen side downward). Then 25 g of filter aid are slurried in 200 to 300 ml pure water and completely transferred into the lab filter. The lab filter is attached to the elevated water tank and purged. After one minute 500 ml water are removed via a filter and then the time for the next 100 ml filtrate stopped. The water value follows from the stopped time as follows:

$$\text{water value} = \frac{480}{\text{time in minutes}}$$

If the result is a water value of less than 150, the determination is done as above, but with the use of only 4 g of filter aid. Then the result is:

$$\text{water value} = \frac{76.8}{\text{time in minutes.}}$$

Hence the shorter the time, required for a specific volume of water to flow through the filter layer, the higher is the water value.

TABLE

Product	Moisture content % by weight	Odor	Flavor	Turbidity	Color	Extract Whiteness %	Bulk density g/dm^3	Wet cake height mm/25g	Darcy Value	Incoming flow behavior	Water value min^1/25 g
Reference Lignocel C120	9.0	8	10 (bitter)	8 (yellow)	3.37	58.4	128	78	5.3	good	770
Sample No. 1	3.5	1	2-3 (mild)	1-2 (Colorless)	0.93	34.2	not determined	83	8.0	good	1098
Sample No. 2	5.3	1	2-3 (mild)	2 (Colorless)	1.04	34.1	125	82	7.3	good	1010
Sample No. 3	7.5	5	6 (neutral)	2-3 (almost colorless)	0.98	36.8	131	79	7.8	good	1125

Patent Claims:

1. Filter aid that comprises finely divided plant fiber particles, **characterized in that** the particles have been subjected to a liquid treatment, which removes the sensorially active substances from the plant fibers.

2. Filter aids according to claim 1, **characterized in that** the particles comprise wood particles.

3. Filter aids according to claim 1, **characterized in that** the particles comprise wood fibers.

4. Filter aids according to claim 1, **characterized in that** the particles comprise wood comminution residues.

5. Filter aid according to any one of claims 1 to 4, **characterized in that** the particles have been subjected to a treatment with a dilute alkali solution.

6. Filter aid according to any one of claims 1 to 4, **characterized in that** the particles have been subjected to a treatment with diluted acid.

7. Filter aid according to any one of claims 1 to 4, **characterized in that** the particles have been subjected to a treatment with an organic or inorganic solvent.

8. Filter aid according to any one of claims 1 to 4, **characterized in that** the particles have been subjected to a treatment with water.

9. Filter aid according to any one of claims 1 to 8, **characterized in that** it essentially comprises only wood particles of one and the same type, size distribution and pretreatment.

10. Filter aid according to one of claims 1 to 8, **characterized in that** it comprises at least two fractions of particles comminuted by different processes.

11. Filter aid according to any one of claims 1 to 8, **characterized in that** it comprises at least two fractions of particles comminuted to different dimensions.

12. Filter aid according to any one of claims 1 to 11, **characterized in that** it comprises fractions of particles produced from at least two different starting materials.

13. ~~Filter aid according to any one of claims 1 to 12, characterized in that it comprises other~~ organic or inorganic fractions that do not affect the filtration properties.

14. Filter aid according to any one of claims 1 to 13, **characterized in that** it comprises other filter-active fractions.

15. Filter aid according to any one of claims 1 to 14, **characterized in that** it comprises other mineral fractions.

16. Filter aid according to any one of claims 1 to 15, **characterized in that** it comprises kieselguhr.

17. Filter aid according to any one of claims 1 to 16, **characterized in that** it comprises perlite.

18. Filter aid according to any one of claims 1 to 17, **characterized in that** the mean particle dimension of the ready-to-use filter aid is below 3.0 mm.

19. Filter aid according to any one of claims 1 to 17, **characterized in that** the mean fiber diameter is below 1.0 mm in the case of fibrous particles.

20. Process for producing the filter aid according to any one of claims 1 to 19, **characterized in that** the particles are digested with the treatment solution during a period of action.

21. Process according to claim 20, **characterized in that** the temperature of the treatment solution during the treatment is in the range of ambient temperature.

22. Process according to claim 20 or 21, **characterized in that** the temperature of the treatment solution during treatment ranges from 50-130°C.

23. Process according to any one of claims 20 to 22, **characterized in that** the temperature of the treatment solution during the treatment is below 100°C and the treatment takes place under atmospheric pressure.

24. Process according to any one of claims 1 to 23, **characterized in that** the treatment is conducted with a dilute alkali solution.

25. Process according to any one of claims 1 to 24, **characterized in that** the temperature of the alkali solution during the treatment ranges from 70 to 90°C.

26. Process according to any one of claims 1 to 25, **characterized in that** the concentration of the dilute alkali solution ranges from 2 to 10% by weight, based on the solids content.

27. ~~Process according to any one of claims 24 to 26, characterized in that the alkali solution~~ used is a sodium hydroxide solution.

28. Process according to any one of claims 20 to 27, **characterized in that** the period of action is of such a duration that at most 10% by weight on an absolutely dry basis of the wood constituents are removed.

29. Process according to any one of claims 24 to 28, **characterized in that** the period of action ranges from 5 to 120 min.

30. Process according to any one of claims 24 to 29, **characterized in that** the density during the treatment ranges from 5 to 25%.

31. Process according to any one of claims 20 to 30, **characterized in that** the particles are washed and dried after the period of action.

32. Process according to any one of claims 20 to 31, **characterized in that** the particle size during the treatment is up to 10 mm, preferably from 0.1 to 1.0 mm.

33. Process according to any one of claims 20 to 32, **characterized in that** the water value is set by influencing the grinding in the wet phase (refiner).

34. Process according to any one of claims 20 to 33, **characterized in that** the particles are further comminuted after the treatment and before the drying, simultaneously with the drying or after the drying.

35. Process according to any one of claims 20 to 34, **characterized in that** the particles are graded after the treatment and the drying.

36. The use of finely divided plant fibers that for a period of action have been subjected to a liquid treatment, which removes the sensorially active substances from the plant fibers, as a filter aid.

37. The use of finely divided plant fibers, which have been produced according to any one of claims 20 to 35, as a filter aid.

38. The use according to claim 36 or 37 in beverage filtration, in particular beer filtration.

39. The use according to claim 36 or 37 in food filtration.

40. The use according to claim 36 or 37 in the sector of the cleaning of liquids in the chemicals industry.

41. ~~The use according to claim 36 or 37 in the sector of the cleaning of auxiliary liquids in~~ metalworking.

42. The use according to claim 26 or 37 in the sector of pharmaceuticals and cosmetics.

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Patent claims:

1. Filter aid which comprises finely divided wood particles which have been subjected to a chemical liquid treatment, characterized in that the particles have been subjected to a treatment with a dilute alkali solution at a temperature below 100°C and at atmospheric pressure, which removes the sensorially active substances from the wood particles.

2. Filter aids according to claim 1, characterized in that the particles comprise wood fibers.

3. Filter aids according to claim 1, characterized in that the particles comprise wood comminution residues.

4. Filter aid according to one of claims 1 to 3, characterized in that it essentially comprises only wood particles of one and the same type, size distribution and pretreatment.

5. Filter aid according to one of claims 1 to 3, characterized in that it comprises at least two fractions of particles comminuted by different processes.

6. Filter aid according to one of claims 1 to 5, characterized in that it comprises at least two fractions of particles comminuted to different dimensions.

7. Filter aid according to one of claims 1 to 6,

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characterized in that it comprises fractions of particles produced from at least two different starting materials.

8. Filter aid according to one of claims 1 to 7, characterized in that it comprises other organic or inorganic fractions which do not affect the filtration properties.

9. Filter aid according to one of claims 1 to 8, characterized in that it comprises other filter-active fractions.

10. Filter aid according to one of claims 1 to 9 characterized in that it comprises other mineral fractions.

11. Filter aid according to one of claims 1 to 10, characterized in that it comprises kieselguhr.

12. Filter aid according to one of claims 1 to 11, characterized in that it comprises perlite.

13. Filter aid according to one of claims 1 to 12, characterized in that the mean particle dimension of the ready-to-use filter aid is below 3.0 mm.

14. Filter aid according to one of claims 1 to 13, characterized in that the mean fiber diameter is below 1.0 mm in the case of fibrous particles.

15. Process for producing the filter aid according to one of claims 1 to 14, characterized in that the particles are digested with the dilute alkali solution

during a period of action.

16. Process according to claim 15, characterized in that the temperature of the dilute alkali solution during the treatment is in the range of room temperature.

17. Process according to claim 15 or 16, characterized in that the temperature of the dilute alkali solution during treatment is 50-100°C.

18. Process according to one of claims 15 to 17, characterized in that the temperature of the dilute alkali solution during the treatment is from 70 to 90°C.

19. Process according to one of claims 15 to 18, characterized in that concentration of the dilute alkali solution is from 2 to 10% by weight, based on the solids content.

20. Process according to one of claims 15 to 19, characterized in that the alkali solution used is sodium hydroxide solution.

21. Process according to one of claims 15 to 20, characterized in that the period of action is of a duration such that at most 10% by weight on an absolutely dry basis of the wood constituents are removed.

22. Process according to one of claims 15 to 21, characterized in that the period of action is from 5 to 120 min.

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23. Process according to one of claims 15 to 22, characterized in that the consistency during the treatment is from 5 to 25%.

24. Process according to one of claims 15 to 23, characterized in that the particles are washed and dried after the period of action.

25. Process according to one of claims 15 to 24, characterized in that the particle size during the treatment is up to 10 mm, preferably from 0.1 to 1.0 mm.

26. Process according to one of claims 15 to 25, characterized in that the water value is set by influencing the grinding in the wet phase (refiner).

27. Process according to one of claims 15 to 26, characterized in that the particles are further comminuted after the treatment and before the drying, simultaneously with the drying or after the drying.

28. Process according to one of claims 15 to 27, characterized in that the particles are classified after the treatment and the drying.

29. The use of finely divided wood particles which have been subjected to a treatment with a dilute alkali solution at a temperature below 100°C and at atmospheric pressure, which treatment removes the sensorially active substances from the wood particles, as filter aid.

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30. The use of finely divided wood particles which have been treated according to one of claims 15 to 28 as filter aid.

31. The use according to claim 29 or 30 in beverage filtration, in particular beer filtration.

32. The use according to claim 29 or 30 in food filtration.

33. The use according to claim 29 or 30 in the sector of the cleaning of liquids in the chemicals industry.

34. The use according to claim 29 or 30 in the sector of the cleaning of auxiliary liquids in metalworking.

35. The use according to claim 29 or 30 in the sector of pharmaceuticals and cosmetics.

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Abstract:

The filter aid comprises finely divided plant fibers, which for a period of action have been subjected to a liquid treatment, which removes the sensorially active substances from the plant fibers.